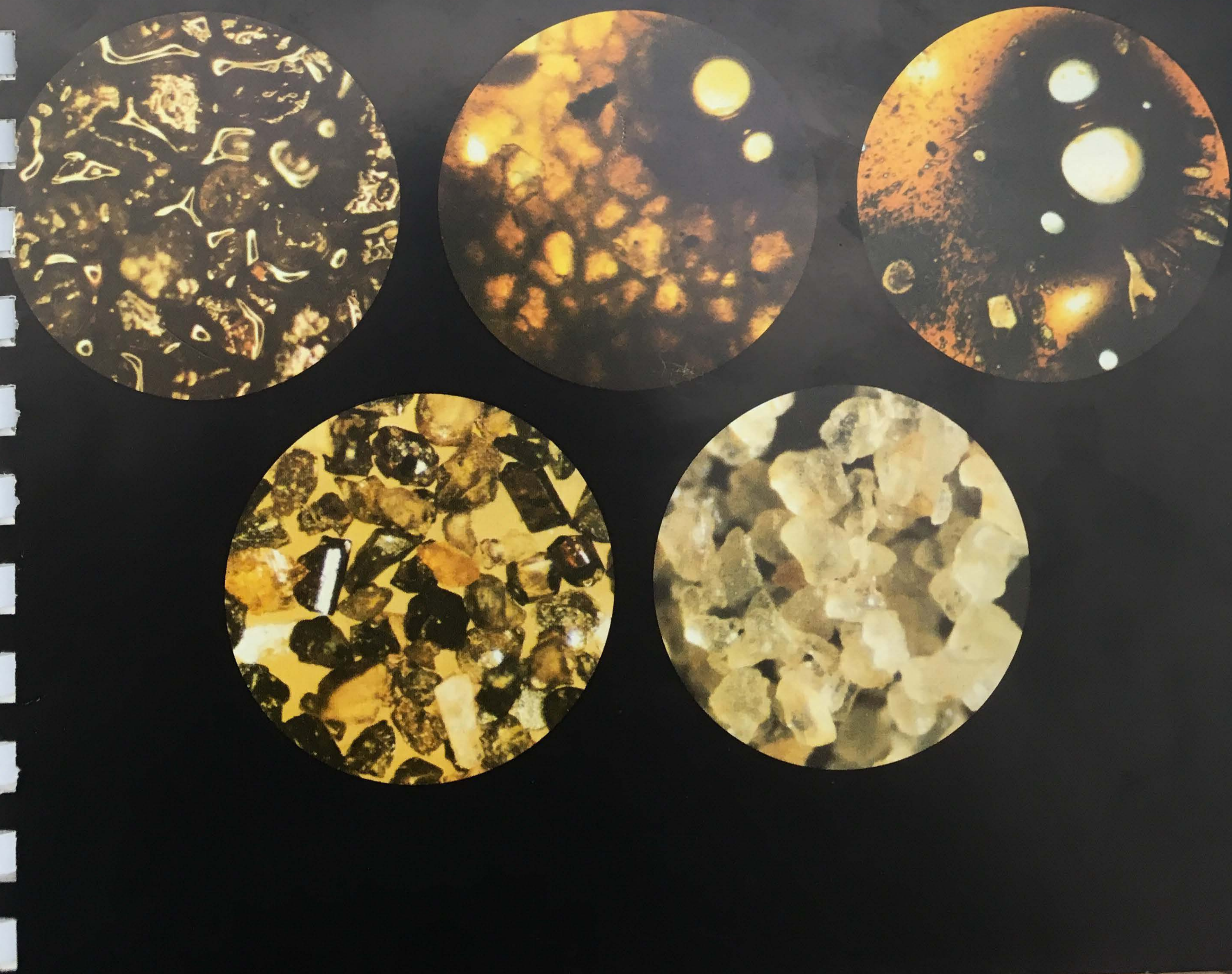
Syncrude

research activities 1974



C O N F I D E N T I A L

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RESEARCH ACTIVITIES 1974

Research Department
Syncrude Canada Ltd.

January, 1975

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1. I N T R O D U C T I O N

1. INTRODUCTION

1.1 Research Objectives

Syncrude's corporate objectives were listed in "Research and Development: A Proposal" (1) and are repeated here (Table 1). The primary departmental technical objectives are listed in Table 2.

Objectives of specific projects are stated in the summary write-ups associated with the details of "Research and Development: A 5-Year Plan". (2)

Specific departmental objectives have been developed for each major functional area -

1. Applied Research

(a) Applied Chemistry

1. To develop a comprehensive knowledge of the chemistry of the bitumen contained in tar sand and that which is recovered through extraction and froth treatment.
2. Specialization in the colloid and surface chemistry of hydrophobic mixed-clay suspensions in water containing organic compounds leading to improvements in processes, reduction of sludge volume and improved water clarification.
3. Research the physical chemistry of water, solutions of electrolytes and interactions with organic compounds leading to an understanding of interactions in extraction and of changing properties over the life of the plant water reclaim system.
4. To continue analysis and characterization of heavy oils and residua, to facilitate the understanding of primary processes.
5. To develop technical competence in the catalytic hydrogenative upgrading of products from bitumen conversion processes to insure availability of adequate technical services for the commercial plants.

TABLE 1

CORPORATE OBJECTIVES

Primary

1. To determine the most economic and technically feasible method for production of synthetic crude oil from Syncrude leases in the Athabasca tar sands.

2. To design, construct and operate such facilities as will realize this purpose.

3. To develop technology to enhance the conservation, recovery and use of the hydrocarbon and mineral resources associated with the Athabasca tar sands.

4. To carry out these objectives in a manner which will recognize legitimate public interests as well as those of the participating shareholders.

Secondary

5. To develop and staff a flexible organization possessing the necessary experience and skills to efficiently accomplish the general corporate objectives.

6. To develop within Syncrude, the capability to observe and recognize the changing scene and to respond with the on-going development of an effective long range plan.

TABLE 2

DEPARTMENTAL OBJECTIVES

To develop and maintain a research and development program including:

1. A balanced approach with complementary activities in research, development, environmental affairs and technical services.

2. A planned sequence of events supporting the start up of the Mildred Lake Plant and anticipating subsequent major plant expansions or new projects.

3. An organizational structure, staff and facility capable of responding to deliberate change with continuing productivity and efficiency.

4. Designated responsibilities and functions fully integrated with Syncrude's engineering projects and operations activities.

5. An Alberta based effort responsive to the unique circumstances and opportunities represented by commercial development of the tar sands.

6. Specialization in hydrocracking of bitumen to allow high liquid yield hydrocracking to become incorporated in future tar sands plants.
7. The high sulfur and nitrogen content of bitumen and its low distillate yield require that a high level of awareness of and active contribution to developments in desulfurization denitrogenation and residue conversion.
8. Research in the physical chemistry, thermodynamics and phase relationships of heavy oils and residua leading to confidence in the scale-up of hydrocracking and in the operability of equipment which handles bitumen or residue at elevated temperatures.

(b) Analytical Development

1. To ensure that the company's technology in analytical chemistry is effectively utilized and kept up-to-date by recruitment and training of competent staff and a continuous review and adaptation of new advances in analytical chemistry developed by the Research Department or found in the literature.
2. To maintain and upgrade laboratory facilities as required to meet departmental objectives.
3. To co-ordinate analytical work done outside the department taking advantage of special expertise found elsewhere.
4. To maintain close liaison with the Mildred Lake Project control laboratory.
5. To develop, test, and implement automated analyzers needed for proper operation of a tar sands complex.

(c) Diversification Studies

1. To develop technology for the conservation, recovery and use of mineral by-products associated with the extraction of synthetic crude oil from the Athabasca tar sands.
2. To seek out and evaluate opportunities to enhance the conservation, recovery and use of fuel resources (methane, coke, synthetic fuel gas, residual oil) which are by-products of processing of the tar sands.

3. To provide for the conservation and recovery of hydrocarbon raw materials which may be utilized as a source of petrochemicals.
4. To assess, develop and provide technology for the production of raw materials (caustic soda, etc.) required in the processing of the tar sands.

(d) Geological Studies

1. To examine all Syncrude Leases in detail to assist in selection of the best mining locations. This will include environmental interpretations, using information from logs, cores, outcrop and previous work by participants, government and private individuals.
2. To undertake a program resulting in better understanding of tar sand processibility and mineral recovery.
3. To identify potential problems resulting from otherwise undetected geological features.

2. Engineering Research

1. Conduct technical and economic feasibility studies on new processes and/or process improvements.
2. Undertake pilot plant investigations in order to demonstrate new technology and to provide the engineering data required in the design and implementation of such technology.
3. Provide liaison between the commercial plant and the Research Department in terms of identifying problem areas requiring laboratory or pilot plant investigation.
4. Maintain an active company-wide patent program.

3. Environmental Affairs

1. To fully implement Syncrude's Environmental Policy.
2. Conduct studies to forecast, define and assess the environmental effects of all stages and aspects of the Company's operations.
3. Inform management of government environmental regulations and policies and anticipate new developments.

4. Develop a Company response to the environmental requirements of the government and the public with due regard for project economics. Confer with management, scientific authorities and governmental agencies; prepare reports and briefs outlining progress, plans and recommendations on environmental matters.
5. Establish procedures to monitor actual environmental impact of the commercial operations for response to governmental and public informational requirements and for use in planning future commercial ventures.

4. Analytical and Technical Services
   1. Provide and co-ordinate analytical, technical and information services that will aid in the achievement of all research and environmental project objectives.
   2. To make available the resources of the group as required by all company departments.
   3. In information services to seek out and supply technical information relevant to the projects in research, development and environmental affairs, and to store such information so that it may be readily retrieved.
   4. To maintain and operate a reserves evaluation laboratory to provide analytical data to establish reserves estimates for all leases plus information required to determine grade distribution, water sand definition and geological information for mine design and tar sand processibility.

5. Research Operations
   1. Provide facilities and operating services, and administrative, clerical and commercial support to the Research Department.
   2. Provide engineering, trades and labour support to maintain and operate the physical research plant.

References

1. *Research Department. Research and Development: A Proposal. January, 1974. 15 pp.*

2. *Research Department. Research and Development: A Five Year Plan. May, 1974, 75 pp.*

1.2 Research Department Organization

In response to the circumstances in which Research has been required and the nature of the work to be undertaken, the Department has been structured by function*:

- Research
  - Applied Research
    - Applied Chemistry
    - Analytical Development
    - Diversification Studies
    - Geological Research
  - Engineering Research
    - Technical Assessment
    - Process Development
    - Incentive Assessment
  - Environmental Affairs
    - Environmental Engineering
    - Ecological Studies
    - Special Projects
    - Environmental Information Services
  - Analytical and Technical Services
    - Analytical Services
    - Reserves Evaluation
    - Environmental Field Monitoring
    - Information Services
    - Data Processing
    - Special Projects
  - Research Operations
    - Engineering Services
    - Operating Services
    - Maintenance Services
    - Business Services
    - Safety and Security

Activities within this functional framework have been organized by project. The research documented in this report is presented on a project basis.

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*A staff directory is included in this report as Section 12.

1.3 Reporting of Activities

This documentation of research activities for 1974 is intended to be a source document for convenient reference for work completed during the year. Detailed reports are issued as a series of interim, progress and subject reports during the course of the year. These reports, most of which are listed in the bibliography, are herein referred to by footnote. This should allow convenient access to the source documents.

Frequent reference is made to the Research Department's monthly progress reports. These reports are intended to allow prompt distribution of preliminary results of investigations and to report the development of on-going projects. The progress reports are intended to be working documents for internal use only.

Research Activities 1974 has been prepared by the Research Department as a permanent record.

2. TAR SAND STUDIES

2.1 Chloride in Tar Sand: Lease 17

A pilot plant study was undertaken to determine the effect of chloride on tar sand processibility (1). As part of this study, it was necessary to develop an analytical method to determine the chloride content of the several different process streams in order to calculate, in turn, a chloride balance around the unit. An analytical method based on the use of a chloride ion electrode was developed (2,3). To establish confidence in the technique, six core holes from the 1974 coring program were analyzed to compare the ion electrode technique with the conventional silver nitrate titration technique. The ion electrode technique proved to be suitable for monitoring chloride in tar sand.

The analytical study revealed an unexpected result in that concentration as high as 2500 ppm chloride ion were found in core hole samples, particularly in high silt and clay containing sample intervals. The scope of the study was then expanded (3) to include six additional core holes selecting holes that might reveal more meaningful results in terms of correlating chloride concentrations with lithological descriptions and hole locations.

In summary, the work led to the following conclusions (4):

1. The chloride content of the core samples analyzed generally increases down hole.
2. The chloride content of silt and clay units increases down hole but does not increase down hole in tar sand units.
3. The chloride content of tar sand units varies considerably from hole to hole.
4. Regionally the weighted averages of chloride concentrations in core holes increased towards the west of the mine area (see map in reference (4)). This conclusion supports the work done by Sproule and Associates (5).
5. Chloride "lows" seem to correspond to basal Devonian limestone "lows".

These conclusions and the chloride data reported in references (1,2,3,4,6) are expected to be useful in updating the plant water balance calculations (7) in predicting the effect of salinity on tar sand processability (8) in elucidating potential processing problems (9), and in assessing the effect of chloride on pond water chemistry and environmental impact (10).

R.E. Hoyle

References

1. Lane, S.J. "Effect of sodium chloride and saline mine water on extraction circuit". Extraction Pilot Plant - Special Report, E-9. June 20, 1974.
2. Research Department Progress Report, September 1, 1974, p. 36.
3. Research Department Progress Report, October 1, 1974, p. 40.
4. Research Department Progress Report, December 1, 1974, p. 51.
5. Sproule, J.G. and Assoc. "Regional hydrogeological study, McMurray Oil Sands area, Alberta: Phase 1. Prepared for the Oil Sands Environmental Study Group, October, 1974, p. 23.
6. Research Department Progress Report, August 1, 1974, p. 41.
7. "Chloride concentrations in saline waters". Syncrude Interoffice Correspondence, ref. no. 19661, T. Voksepp to G.R. Gray, November 14, 1974.
8. Research Department Progress Report, September 1, 1973, p. 24.
9. "Phone conversation with J. Robertson of ER&E concerning effect of saline mine water" Syncrude Interoffice Correspondence, ref. no. 6212, W. Prior to G.R. Gray, May 23, 1974.
10. Research Department Progress Report, October 1, 1974, p. 37.

2.2 Effects of Daylight on Tar Sand Aging

In almost all cases observed, aged tar sand gives lower recoveries and poorer froth qualities in the hot water process than unaged material. Aging of a tar sand causes changes in some of its constituents and occurs on weathering. This is confirmed by aging effects on processibility decreasing with depth in a pile of tar sand, from the surface into the interior (1). Although changes in the inorganic constituents of tar sands can result from aging (e.g., a pH change) it is also likely that the organic constituents are susceptible to weathering conditions. The ease of oxidation of various organic compounds,

especially those containing sulfur, has been demonstrated in several Syncrude Research documents (*1*).

The initiation of chemical or physical changes in a complicated system such as tar sand can be attributed to many factors, among which the following may be cited:

1. Contact with atmospheric oxygen.
2. Gradual loss of connate water by evaporation, resulting in the concentration of water soluble species (acids, bases, salts and surfactants).
3. Presence of catalytic solid surfaces.
4. Photochemical reactions through exposure to daylight.

Photochemical reactions can be initiated by exposing the tar sand to daylight and can contribute appreciably to the aging phenomenon. A variety of such reactions can be induced in the presence of oxygen, optimum pH media, and catalyst (e.g., solid surfaces) and for this reason some controlled irradiation experiments should be conducted on tar sand and bitumen components. An introduction to the variety of possible photochemical reactions and of their potential is given in reference (*2*).

<u>Future Activities</u>

Exposure of a variety of tar sands and some bitumen components to an intense visible and ultraviolet light will be conducted and effects on the following will be monitored:

1. Response to hot water processing.
2. Chemical changes in the bitumen components.
3. Collection and characterization of gases evolved.

L.H. Ali

<u>*References*</u>

1. *Research Department Research Activity, 1973. January, 1974, p. 5*
2. *Research Department Progress Report, September 1, 1974, p. 3.*

3. PRIMARY EXTRACTION

3. PRIMARY PROCESSING

3.1 Extraction Piloting

3.1.1 15 TPH Pilot Plant

The 15 TPH pilot plant completed, during the year, its original objective of generating design data for the commercial plant. Approximately 6,000 tons of tar sand, representing only 400 operating hours, were processed during 1974.

Sampling, material balancing and reporting procedures remained unchanged from that reported in 1973 (*1*).

In addition to the investigations summarized in this Research Activities report, the pilot plant produced feedstock for the dilution centrifuge pilot plant programs.

Although the 2.5 TPH experimental extraction circuit (see Section 9.1) will be used for future extraction programs, the 15 TPH pilot plant will be maintained on an operational basis.

G.R. Lorenz

Reference

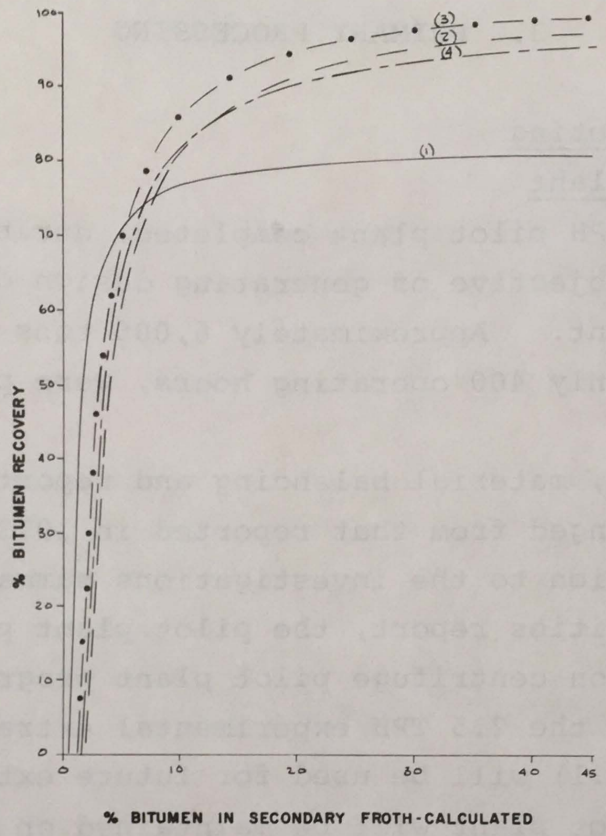
1. *Research Department Research Activity, 1973, January, 1974. p.7.*

3.1.2 Analysis of 15 TPH Extraction Unit Performance

Additional pilot plant work formed the basis for three reports (*1,2,3*) dealing with the design and operation of the secondary froth cleaner, a performance comparison of Maxwell and Denver flotation cells and the processing of high fines tar sand. A fourth report analyzing the particle size fractions around the circuit is in preparation (*4*). Several pilot plant tests were undertaken to examine the effectiveness of froth underwash in the primary separation vessel (*3*).

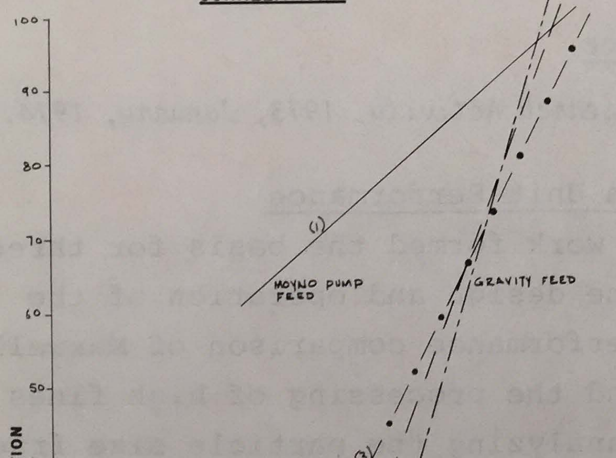
COMPARISON OF % BITUMEN RECOVERY CORRELATIONS

FIGURE 1.



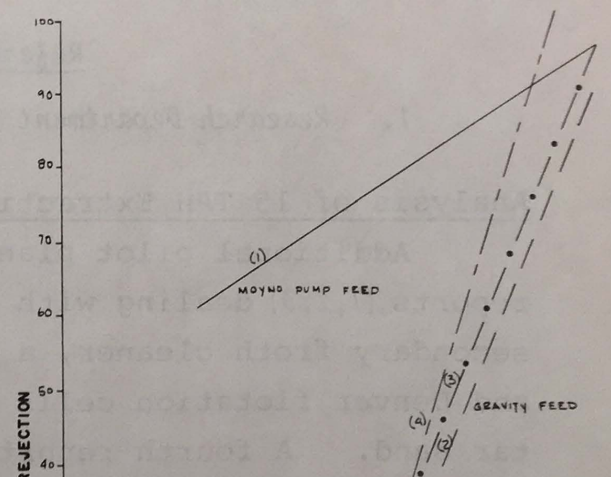
COMPARISON OF WATER REJECTION CORRELATIONS

FIGURE 2.



COMPARISON OF SOLIDS REJECTION CORRELATIONS

FIGURE 3.



The following summaries indicate the major findings of these investigations.

1. Secondary Froth Cleaner

Froth cleaner performance could not be related to loading or residence time variables for either the 12 or 24 inch diameter units; however, the 12 inch cleaner could not cope with the volume of secondary froth produced from a high fines tar sand. The suggested mechanical design criteria were therefore froth loading of about 1000 lb/hr-ft^2, and retention time of 15 minutes.

The main factors found to influence bitumen recovery, and solids and water rejection within the froth cleaner, were the feed methods (Moyno pump vs. gravity) and the secondary froth composition. Cleaner size and tar sand type were relatively unimportant. Figures 1 through 3 illustrate these dependencies by comparing correlations from four different sets of data.

Data Set and Curve No.	Cleaner Diameter Inches	Feed Method	Tar Sand Type
1	24	Moyno Pump	Average
2	24	Gravity	Average
3	12	Gravity	Average
4	24	Gravity	High Fines

Bitumen which reports to the cleaner underflow can be recycled to the inlet of the secondary cells where the recovery is comparable to that of middlings stream.

FIGURE 4.

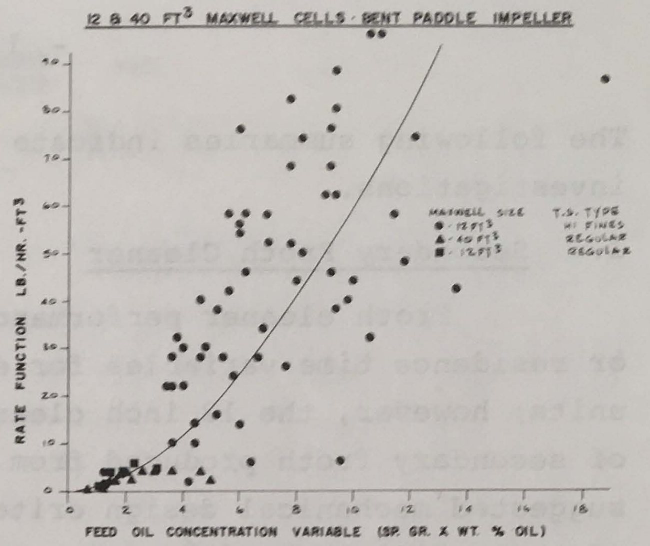


FIGURE 5.

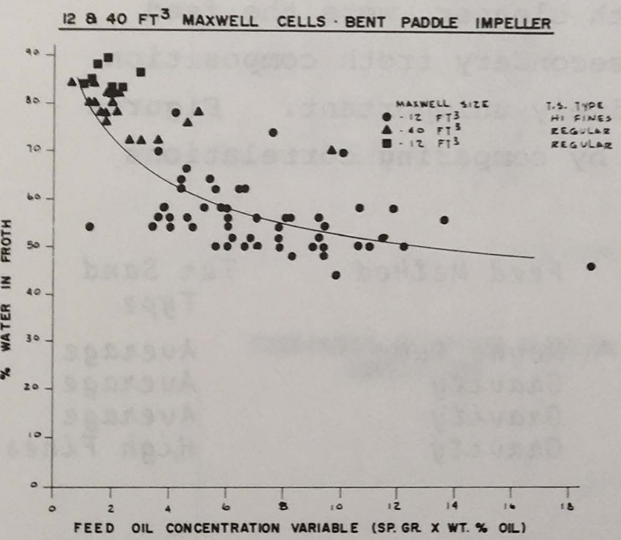
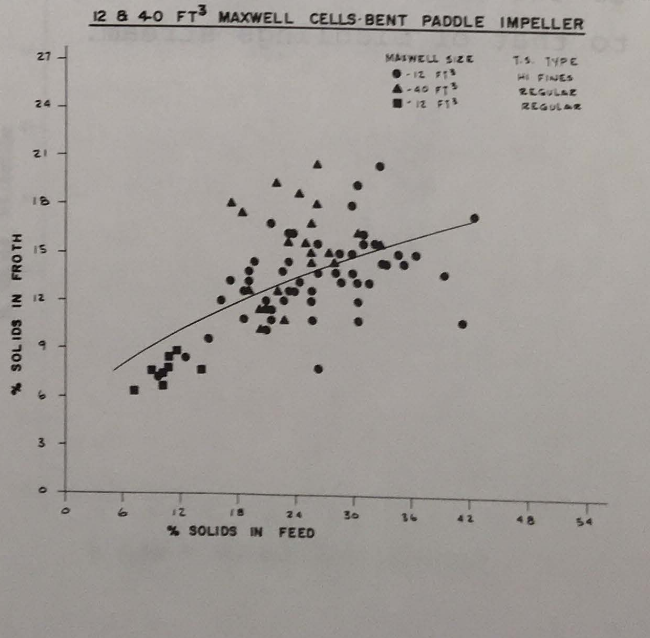


FIGURE 6.



2. Maxwell and Denver Cell Performance in the Secondary Recovery Circuit

An extensive amount of pilot plant data formed the basis for assessing the performance of 12 and 40 cu. ft. Maxwell cells and the individual 12 cu. ft. Denver cells. Cell performance was quantified in terms of froth composition and a rate function which represented the pounds of oil recovered as froth per hour in one cubic foot of cell. This rate and the composition of the froth could be related to the oil and solids content of the cell feed. Figures 4 through 6 show these relationships for the 12 and 40 cu. ft. Maxwell cells. The correlations are independent of cell size and tar sand type. Figures 7 and 8 present the rate function and froth solids correlations for the Denver cells. These relationships are the same for regular and high fines tar sand. However, the water content of Denver cell froth is a function of tar sand type as shown in Figures 9 and 10.

An overall comparison of these results reveals that Maxwell cells recover more oil than a Denver cell of equal volume provided the feed is above 3% bitumen by weight. Below this value Denver cells are superior. Froth produced by the Maxwell cell is generally wetter but has the same solids content as Denver froth. Therefore, the Maxwell cell is a viable alternative to the Denver cell and selection of the preferred secondary circuit configuration must consider the associated capital and operating costs for a given oil recovery. Since the Maxwell cell performs best with a high oil content in the feed, circuits employing Maxwell cells followed by Denver cells were considered and have been incorporated in the commercial design. The correlations developed permitted the sizing and staging calculations required in evaluating such alternatives.

FIGURE 7.

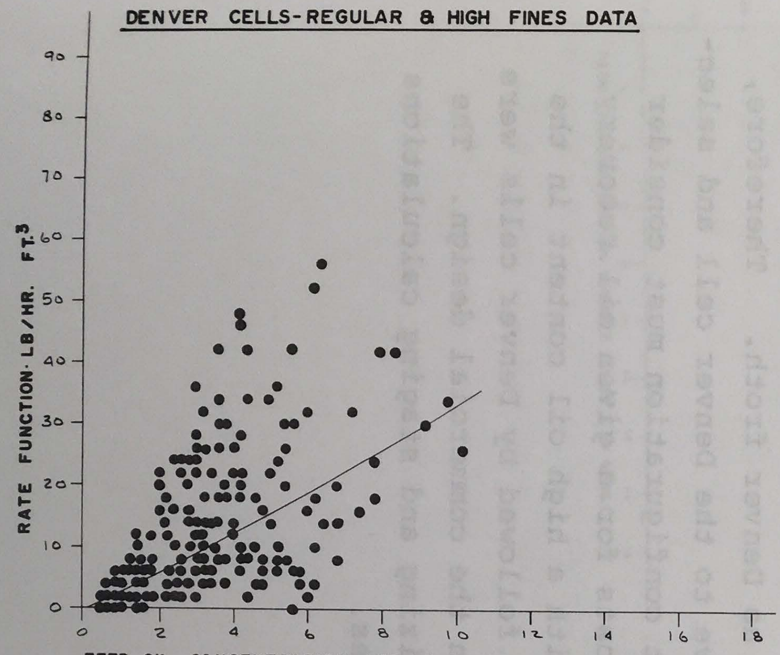


FIGURE 8.

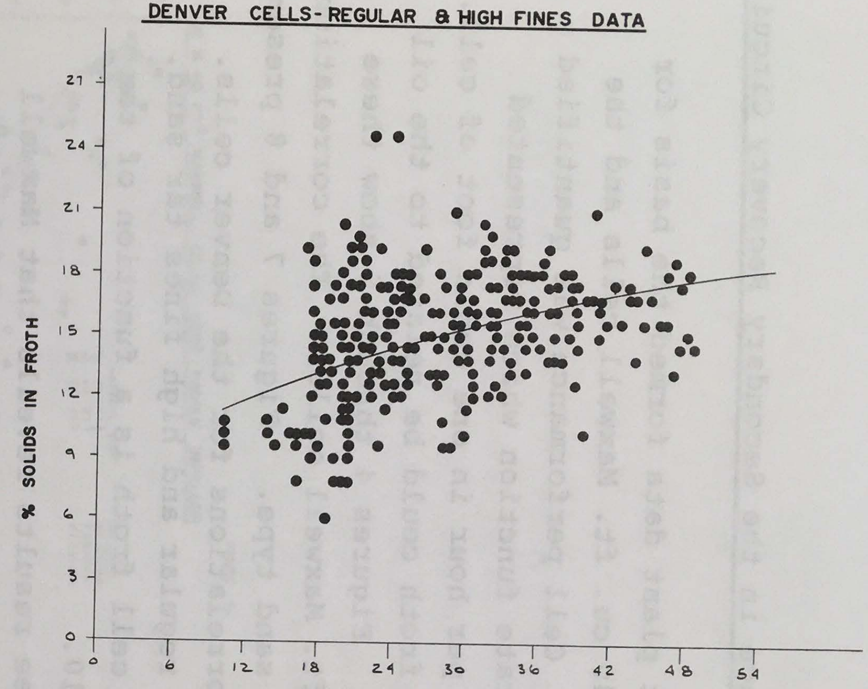


FIGURE 9.

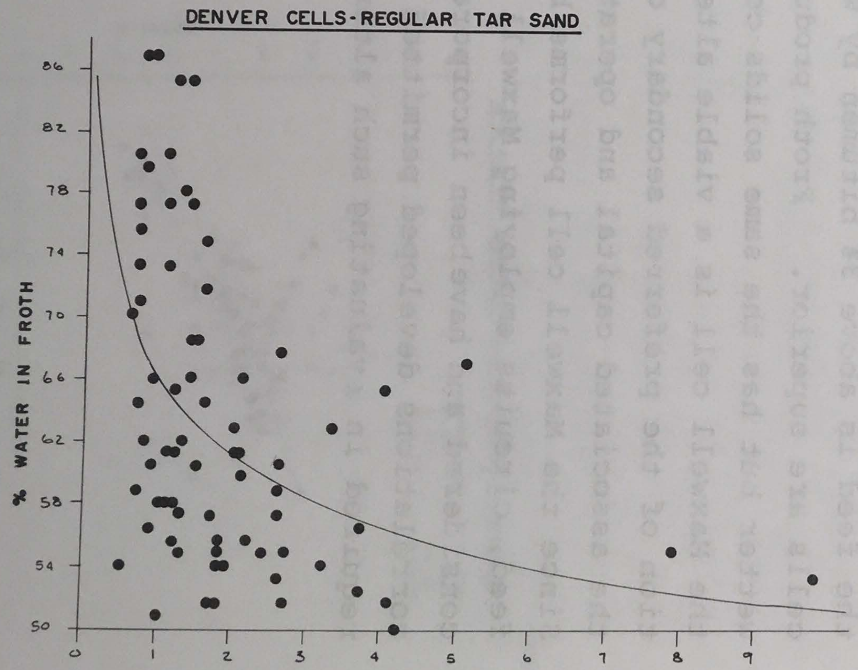
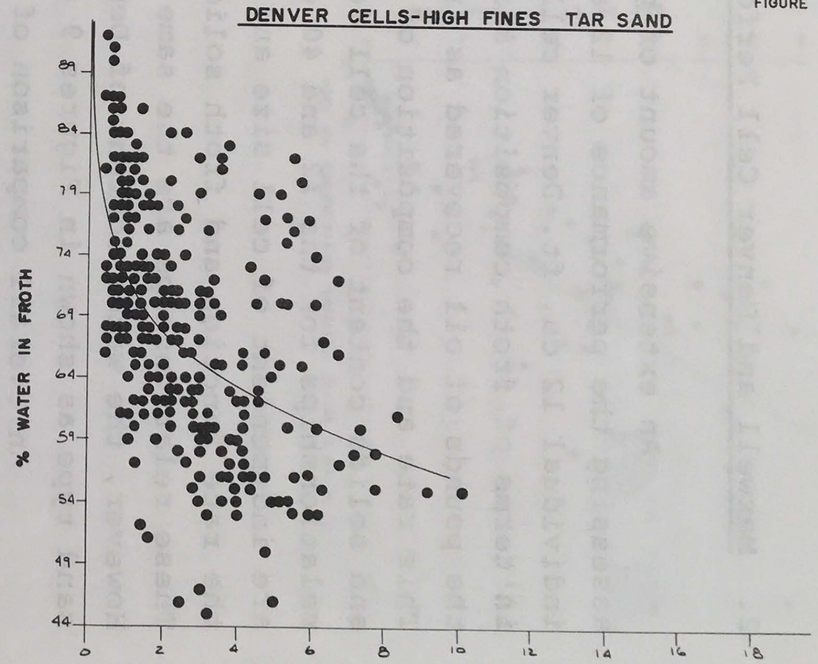


FIGURE 10.



3. Processing of High Fines Tar Sand

Significant quantities of high fines material had never been run in the 15 TPH pilot unit. Therefore some 5000 tons of tar sand having an average bitumen content of 8.0% (range 6.0 to 10.9%) with 20% of the solids less than 325 mesh (range 9.9 to 35.6%) were processed in order to establish a set of operating conditions which allowed sustained operation. With average tar sand, the primary tails withdrawal rate has been controlled from a sand rake torque measurement. However, this type of control led to erratic performance, so control of primary tails density was required during the processing of high fines tar sand. The circuit required 85 lb water/100 lb tar sand which represents an increase of 20 lb/100 lb tar sand over that needed for regular material. No advantage was realized from pH control and a fixed caustic addition rate of 0.035 lb/100 lb tar sand proved adequate. Under these conditions average recoveries and froth qualities are as shown in Table 3, typical values for regular tar sand are included for comparison.

TABLE 3

		High Fines	Regular
Primary Recovery		55	86
Secondary Recovery		30	7
Total Recovery		85	93
Primary Froth	O	49.0	66.4
	W	44.5	24.7
	S	6.5	9.9
Secondary Froth	O	31.9	23.8
	W	55.8	58.7
	S	12.3	17.5

TABLE 4

AVERAGE PARTICLE SIZE FRACTIONS

	0-5	5-10	10-20	20-30	30-44	-44	44-74	74-147	+147 Microns
Average Tar Sand									
Tumbler									
PSV Feed	84.62	93.35	91.80	87.54	86.90	89.31	83.41	95.80	98.26
Reject	15.38	6.65	8.21	12.46	13.10	10.69	11.59	4.20	1.74
Primary									
Froth	7.79	7.48	5.84	8.04	4.50	6.07	4.74	1.49	0.14
Middlings	38.17	48.20	52.95	49.75	35.55	41.66	21.10	11.43	3.50
Tailings	54.04	44.32	41.20	42.21	59.96	52.27	74.15	87.08	96.30
Secondary									
Froth	16.99	11.94	8.98	5.86	2.29	9.62	4.25	2.46	1.91
Tailings	83.01	88.06	91.02	94.14	97.71	90.38	95.75	97.54	98.09
Cleaner									
Froth	52.25	45.71	39.68	37.32	38.47	45.42	45.01	52.19	49.56
Tailings	47.76	54.29	60.32	62.68	61.53	54.58	54.99	47.81	50.44
High Fines Tar Sand									
Tumbler									
PSV Feed	81.13	75.17	75.06	79.23	86.19	80.65	85.32	92.34	97.88
Reject	18.87	24.83	24.94	20.77	13.81	19.35	14.68	7.66	2.12
Primary									
Froth	4.80	2.56	3.20	3.53	2.80	3.49	3.12	0.57	0.04
Middlings	29.86	56.53	39.14	42.84	40.73	38.91	34.68	28.97	14.85
Tailings	65.34	40.92	57.66	53.63	56.48	57.60	62.20	70.46	85.11
Secondary									
Froth	19.95	7.53	22.93	10.37	8.17	13.80	7.93	2.89	1.39
Tailings	80.05	92.47	77.07	89.03	91.83	86.20	92.07	97.12	98.61
Cleaner									
Froth	59.78	57.36	52.68	52.91	52.13	54.53	48.41	48.82	53.52
Tailings	40.22	42.64	47.32	47.09	47.87	45.47	51.59	51.18	46.48

4. Particle Size Splits Around Circuit

Suites of samples around the extraction circuit provided information on the solids particle size distribution in the various streams. These data in conjunction with the stream assays and flow rates enabled calculation of the splits experienced by the various size fractions in each unit. Table 4 indicates some average values for regular and high fines tar sands. It appears from these results that consideration of only two size fractions, namely +44 and -44 micron, would adequately characterize the partitioning of solids between various streams within the process.

5. Primary Froth Underwash

Tests carried out during the high fines processing work failed to confirm that underwash brought about any reduction in the solids level of primary froth.

S.J. Lane
G.R. Lorenz
N.N. McRae
L.J. Falkenberg
K.C. Porteous

References

1. "Secondary Froth Cleaner", Extraction Pilot Plant Special Report E-7, S.J. Lane and L.J. Falkenberg, March 21, 1974.
2. "Maxwell and Denver Cell Performance in the Secondary Recovery Circuit", Extraction Pilot Plant Special Report E-8, G.R. Lorenz, N. McRae and K.C. Porteous, June 1974.
3. "Processing of High Fines Tar Sand", Extraction Pilot Plant Special Report E-10, G.R. Lorenz and K.C. Porteous, July 5, 1974.
4. "Particle Size Splits Around the Extraction Circuit", S.J. Lane, G.R. Lorenz, N. McRae (in preparation).
5. "Commercial Froth Cleaner and Secondary Circuit Design" Letter from W.J. Lavender to W. Daly, Canadian Bechtel, Reference No. 1082, February 5, 1974.

3.2 Slurry Distribution and Cell Dynamics

A significant concern in the design of the commercial primary separation vessels relates to the distribution of slurry across these units. In this regard there are conflicting objectives. The bitumen particles, which ultimately report to the primary froth and the middlings phase, should be dispersed uniformly across the entire vessel, whereas the sand particles which form the primary tails should be restricted to the centre of the vessel. If this sand received the same uniform distribution required for the bitumen and middlings phases, excessive raking requirements would result. Therefore, three separate studies were undertaken in an effort to gain information relative to the distribution of material and the flow patterns within the primary vessel. This information also provides a useful starting point for a more detailed examination of the processes occurring within the cell.

3.2.1 Solids and Bitumen Distribution

Froth loading was calculated by measuring the time for a particular volume of water to be displaced by froth in a closed top glass tube. Measurements were taken at various locations throughout the froth formation zone of the vessel. The sand loading was calculated by placing a rack of 6 test tubes in various radial locations at the bottom of the vessel (1). The hinged cover sealing the test tubes was opened for a specified period of time. The rack was then removed and the sand level in each test tube measured.

The results of the tests (2) indicated that half of the froth is formed at the outside 50% area of the vessel and half of the sand is deposited at the inside 25% area of the vessel. Since the sand exits from the centre of the vessel and the froth from the perimeter of the vessel the results show efficient design of the slurry distributor.

G.R. Lorenz

References

1. Research Department, Annual Documentation, 1971, S-3M-71, December 29, 1971.

2. "Solids and Bitumen Distribution of the 5' Dia. PSV". Syncrude Interoffice Correspondence, G.R. Lorenz to K.C. Porteous, June 20, 1974.

3.2.2 Distribution of Bitumen Particles

The discharge of slurry into the primary separation cell is expected to induce significant currents which would serve to distribute bitumen particles across the vessel. In a crude attempt to simulate this situation, mixtures of buoyant plastic particles and dry sand were fed into a tank of water through a funnel which nominally delivered 2250 lbs sand/hr-ft^2 of tank (1). The buoyant plastic particles had a size and specific gravity such that their rise velocity approximated that of bitumen particles. A series of concentric baffles subdivided the liquid surface and kept the floated particles separated. The material collected between successive baffles provided the data necessary to relate particle loadings to radial position.

In some tests a deflection cone was mounted within the tank. Figure 11 shows that the distribution of the plastic particles across the tank is strongly influenced by the position of the deflection cone and a reasonable distribution can be obtained by the action of the slurry jet. The withdrawal of middlings in an actual primary separation cell should further aid the distribution of the bitumen particles.

K.C. Porteous

Reference

1. *Syncrude Research Department Progress Report, July 1, 1974, p. 19.*

3.2.3 Extraction Cell Dynamics

1. Feed Slurry Distribution

Transient temperature response of the 15 TPH primary separation vessel was used to assess the adequacy of feed slurry distribution across the vessel. The experiments were conducted by producing a step increase or decrease in feed temperature and subsequently measuring temperature as a function of time at a number of points within the vessel (1). Temperatures of the effluent streams as a function of time were also measured. While there were marked differences between the heating and cooling

FIGURE 11.

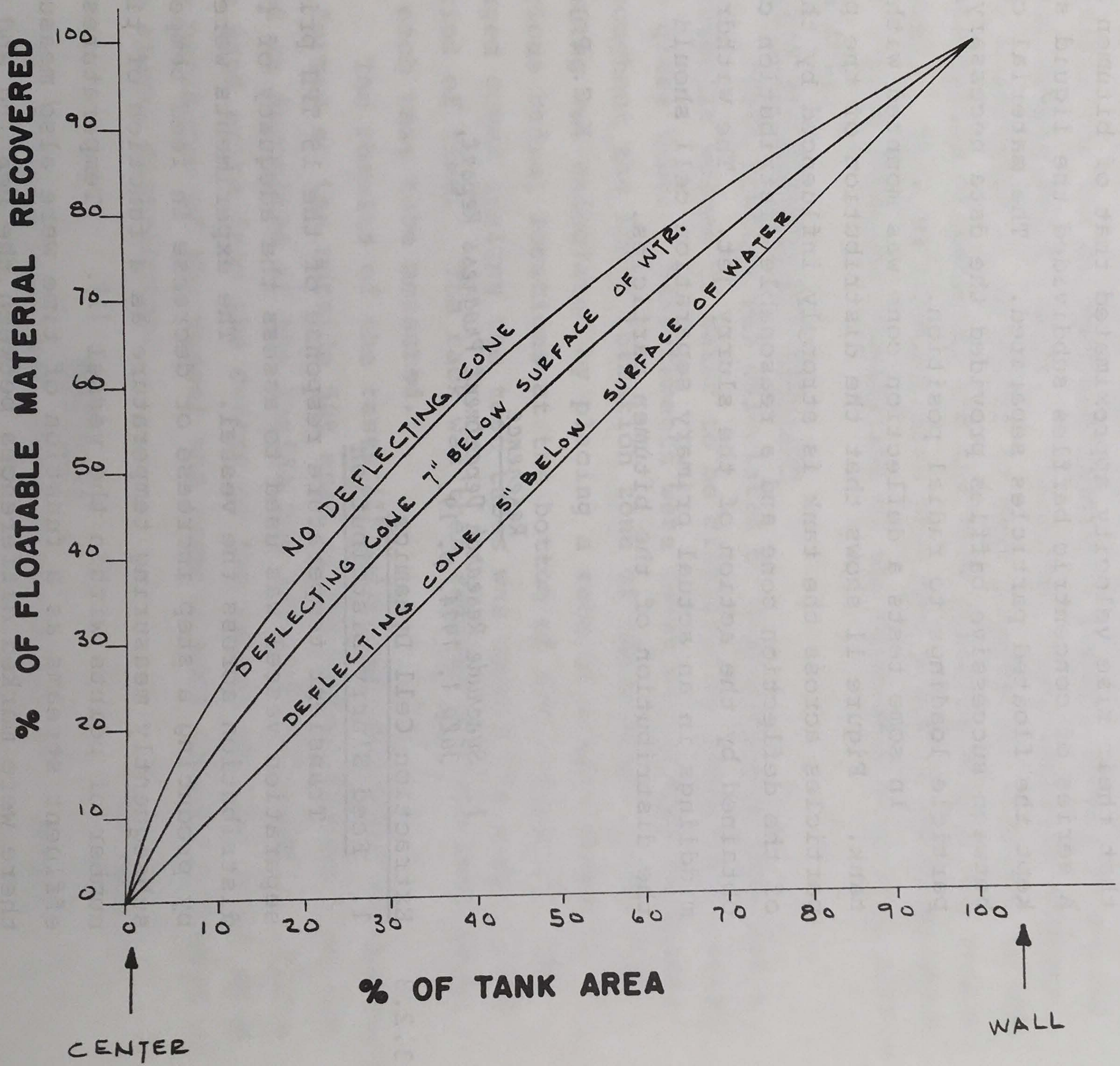
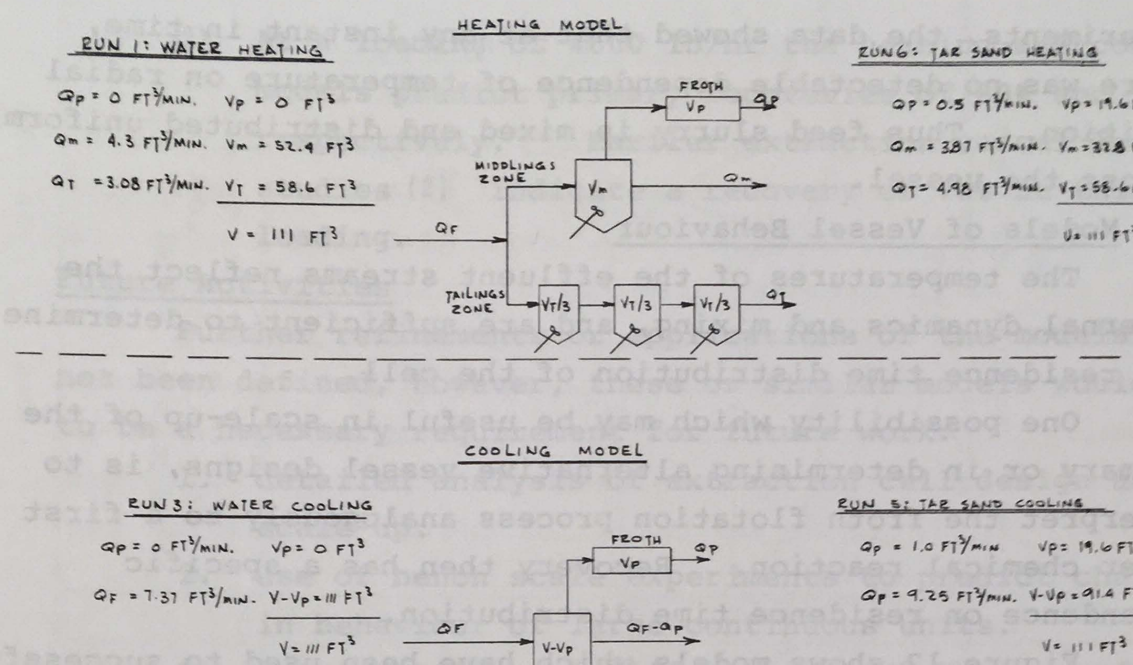
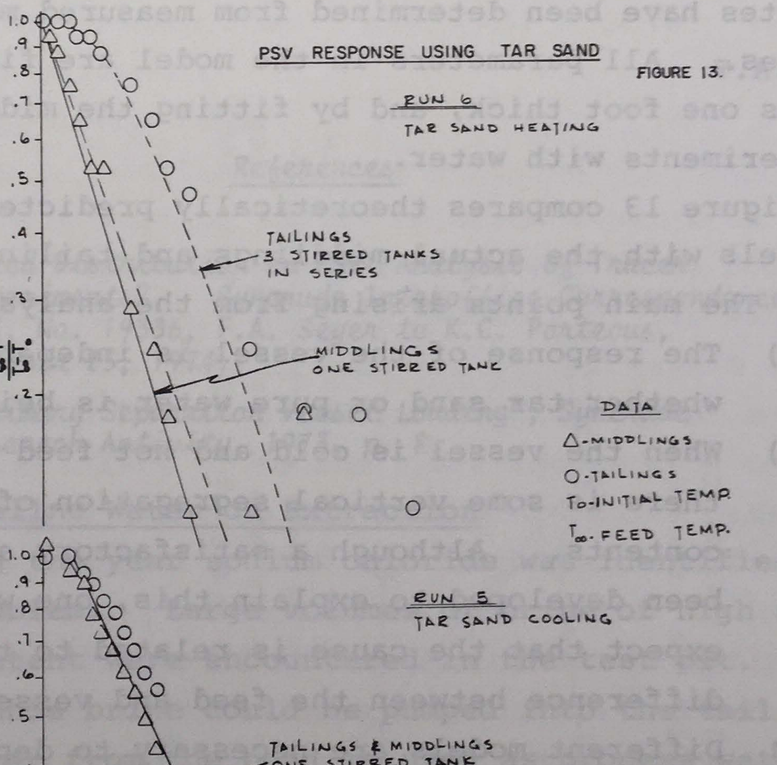

% OF FLOATABLE MATERIAL RECOVERED
0
10
20
30
40
50
60
70
80
90
100
NO DEFLECTING CONE
DEFLECTING CONE 7" BELOW SURFACE OF WTR.
DEFLECTING CONE 5" BELOW SURFACE OF WATER
% OF TANK AREA
CENTER
WALL

FIGURE 12.

MODELS FOR VESSEL RESPONSE





experiments, the data showed that at any instant in time, there was no detectable dependence of temperature on radial position. Thus feed slurry is mixed and distributed uniformly across the vessel.

2. <u>Models of Vessel Behaviour</u>

The temperatures of the effluent streams reflect the internal dynamics and mixing, and are sufficient to determine the residence time distribution of the cell.

One possibility which may be useful in scale-up of the primary or in determining alternative vessel designs, is to interpret the froth flotation process analogously to a first order chemical reaction. Recovery then has a specific dependence on residence time distribution.

Figure 12 shows models which have been used to successfully predict the dynamics of the primary extraction vessel. Volumetric flow rates have been determined from measured mass flow rates and densities. All parameters in the model are fixed by assuming the froth is one foot thick, and by fitting the middlings data from the experiments with water.

Figure 13 compares theoretically predicted response of the models with the actual middlings and tailings temperature data. The main points arising from the analysis and data are:

a) The response of the vessel is independent of whether tar sand or pure water is being processed.

b) When the vessel is cold and hot feed is introduced, there is some vertical segregation of the vessel contents. Although a satisfactory argument has not been developed to explain this, one would intuitively expect that the cause is related to the density difference between the feed and vessel contents.

c) Different models are necessary to depict the heating and cooling experiments. The actual operation of the vessel (isothermal) is somewhere between these two extremes.

d) At a loading of 4600 lb/hr the heating and cooling models predict primary recoveries of 80% and 79% respectively. Earlier extraction vessel loading studies (2) indicate a recovery of 78% at this loading.

Future Activities

Further refinements or applications of the models have not been defined, however, these or similar models would seem to be a necessary requirement for future work:

1. detailed analysis of extraction cell design and scale-up.
2. use of bench scale experiments to predict changes in behaviour of large continuous units.
3. interpretation of differences in behaviour of different pilot units.

F.A. Seyer

References

1. *"Feed Distribution in PSV: Analysis of Tracer Experiments". Syncrude Interoffice Correspondence, Ref. No. 19336, F.A. Seyer to K.C. Porteous, October 25, 1974.*
2. *"Primary Separation Vessel Loading", Syncrude Research Activity, 1973, p. 8.*

3.3 Effect of Saline Water on Extraction

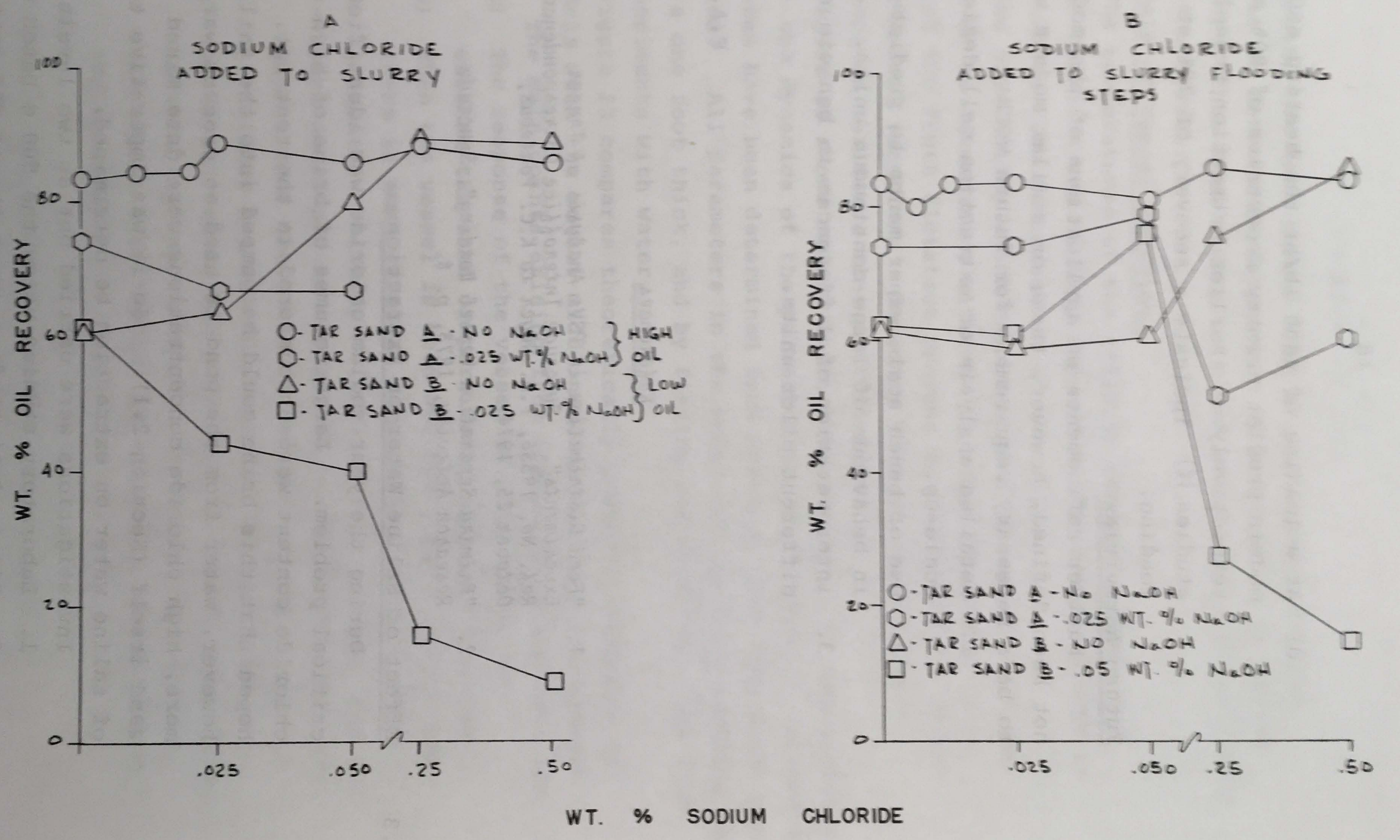
During the year sodium chloride was identified as a critical problem. Large volumes of brine of high sodium chloride content were encountered in the test pit. It was hoped that this brine could be pumped into the tailings pond, however, water from the pond is used as process water. Furthermore, high chloride concentrations were determined in the tar sand itself (Section 2.1). So it was imperative that the effect of saline water on extraction be determined.

Investigations were carried out at two levels:

1. Laboratory studies using the 500 g batch extraction unit.
2. Pilot plant studies using the 15 TPH unit.

FIGURE 14.

EFFECT OF SODIUM CHLORIDE ON THE RECOVERY OF HIGH OIL AND LOW OIL TAR SANDS



3.3.1 Laboratory Batch Extraction Unit Studies

Previous tests in the 500 g batch extraction unit (1) have shown that sodium chloride addition to an average oil tar sand, in dosages of 0.01 to 0.05 weight per cent, was harmful to the process. In combination with sodium hydroxide (0.01 to 0.03 wt %) the effect of the salt was diminished.

Because the sodium chloride levels of the mixtures tested were below those predicted by Bechtel in Plant 5 process water (2) additional work encompassing simultaneous concentrations of sodium hydroxide and sodium chloride of 0.05 to 0.5 wt %, respectively, was carried out on the following tar sands: (1,2,3)

		Weight Per Cent			
		Oil	Water	Solids	-325
A	High Oil - Low Fines	12.84	2.16	84.99	9.95
B	Low Oil - High Fines	8.42	6.88	84.70	18.06
C	Average Oil - Average Fines	9.76	5.54	84.70	11.74

Others Low to Average Oil
Average to High Fines

The most important results achieved are (Figure 14 A and B):

1. The effect of sodium chloride depends on tar sand type. When added in dosages up to 0.5 wt %, sodium chloride does not hurt the oil recovery of tar sand A (2). However, the oil recovery of tar sand B (2) and tar sand C (3) decreased in the presence of sodium chloride.
2. The effect of sodium hydroxide depends on tar sand type.
3. Sodium chloride and sodium hydroxide effects are antagonistic, that is, the effect of one is offset (at least partially) by the other.
4. The effects of sodium hydroxide and sodium chloride depend whether these chemicals are added in the slurry or in both stages (slurry and flooding).

The study of the parameters responsible for sodium chloride effects are being investigated. Some of the findings are reported in Sections 3.5 and 3.6.

Tests to inactivate the sodium chloride effect on some of these tar sands, using humic acid and asphaltene species are described in Section 3.4.7.

Future Activities

Continuation of the study along the lines described in Section 3.4.7 is planned. Some chemicals, other than those of humic acid type, should also be tested. Their choice will be based on colloid-interfacial chemistry studies designed to promote attachment of oil to air, spreading of oil around gas bubbles, separation of oil from solids and water creation of hydrophonic nuclei for formation and coalescence of bubbles, etc., as described in references 4 and 5.

M.V. Baptista

References

1. *Research Department Progress Report, February 1, 1974, p. 12, Appendix 3, Figure 1B.*
2. *Research Department Progress Report, February 1, 1974, p. 12, Appendix 3, Figure 1B.*
3. a) *Research Department Progress Report, February 1, 1974, p. 14, Appendix 3, Figure 1B.*
   b) *Research Department Progress Report, June 1, 1974, p. 16, Table 1.*
   c) *Research Department Progress Report, August 1, 1974, p. 1, Tables 1 and 2.*
   d) *Research Department Progress Report, November 1, 1974, p. 6, Table 1.*
4. *Research Department Progress Report, May 1, 1974, p. 13.*
5. *Research Department Progress Report, June 1, 1974, p. 9.*

3.3.2 Pilot Plant Studies

The effect of salt and saline mine water on extraction was the subject of two pilot plant runs and special reports (1,2).

In the first run sodium chloride was added in dry form to the process, and decreases in froth quality and recovery resulted.

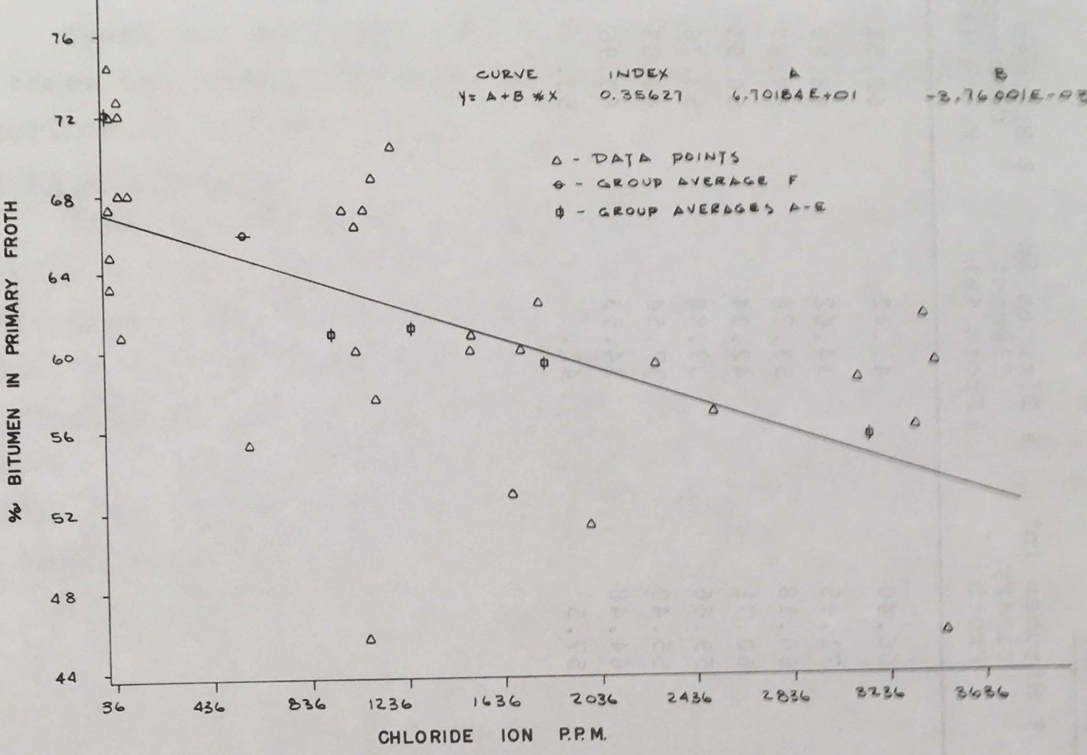
TABLE 5

AVERAGE CIRCUIT PERFORMANCE

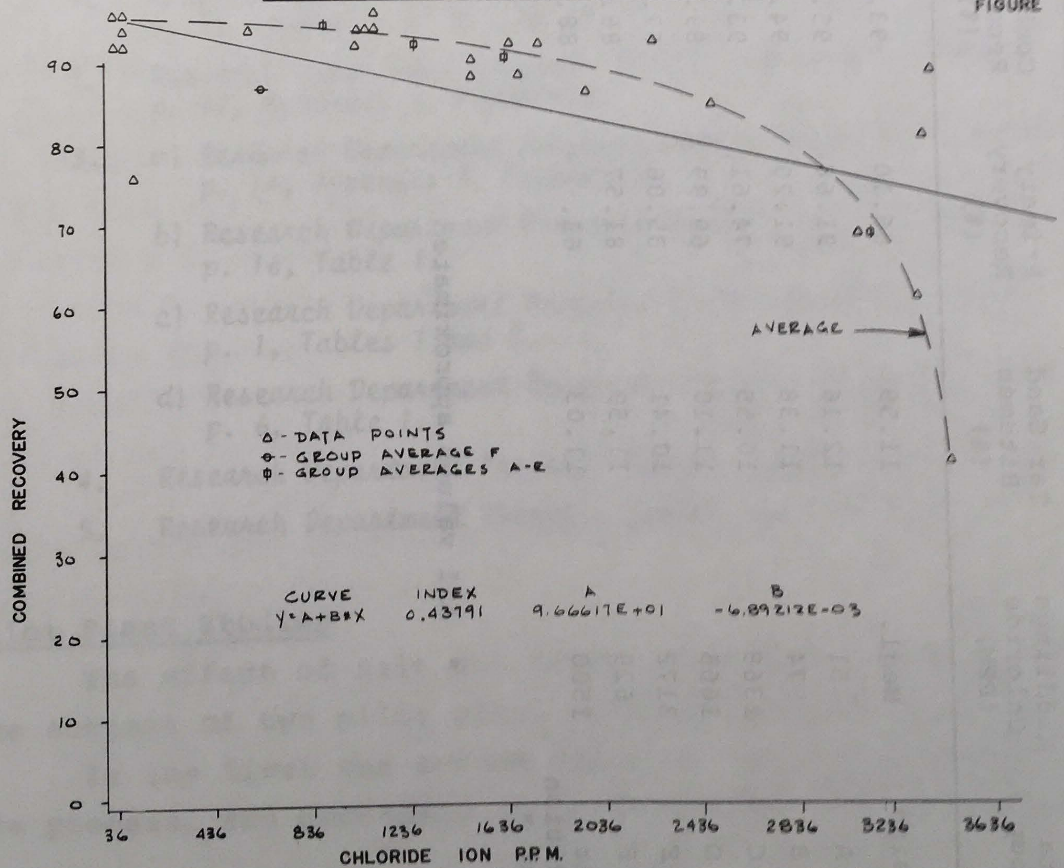
Data Group	Middlings Chloride (PPM)	Tar Sand Bitumen (%)	Primary Recovery (%)	Combined Recovery (%)	% Bitumen in Primary Froth	% Bitumen in Cleaner Froth (%)	% Bitumen in Combined Froth (%)
Design	Negl.	11.59	86.10	93.0	66.40	41.42	62.51
A	51	12.16	91.68	95.40	71.95	34.62	68.95
B	974	11.38	91.20	94.66	60.18	53.74	59.87
C	1368	10.55	74.61	93.23	60.71	42.34	56.95
D	1665	11.10	69.99	89.39	59.66	39.88	53.76
E	3172	10.41	35.06	67.88	55.40	37.54	44.33
F	629	11.59	81.57	86.89	64.48	34.53	60.96
Salt Addition Test *	1500	11.03	68.	88.	57.5	41.	53.

* All values approximate

% BITUMEN IN PRIMARY FROTH VS. CHLORIDE IN P.P.M. FIGURE 15.



COMBINED RECOVERY VS. CHLORIDE ION P.P.M. FIGURE 16



The addition of salt to the circuit caused the chloride ion content of the various streams to increase with time. By treating the primary vessel as a stirred tank, it was possible to obtain an adequate prediction of the observed salt build-up in the middlings. Sodium hydroxide addition was in the range 0.017 to 0.039%.

Some concern was expressed that the salt addition carried out in this manner might not be a good representation of actual mine water. To assess this possibility, 25,000 gallons of mine water was trucked to Edmonton for tests in the extraction pilot plant. The chloride level in this mine water was 7,340 ppm and was blended with city water in various proportions such that the resultant process water would contain about 1,200, 1,900, 3,000, and 3,800 ppm chloride.

In general, the corresponding results of the mine water run were the same as those obtained at steady state in the salt addition tests as indicated in Table 5. In this run sodium hydroxide addition was in the range 0.014 to 0.044%.

The effect on the process is what is referred to in mineral flotation as "reduced selectivity". This means that the bitumen content of primary froth is reduced with salt addition while bitumen in the tailings and middlings stream increased. The net result is thus a reduced froth bitumen recovery for both the primary separation and extraction circuit as a whole.

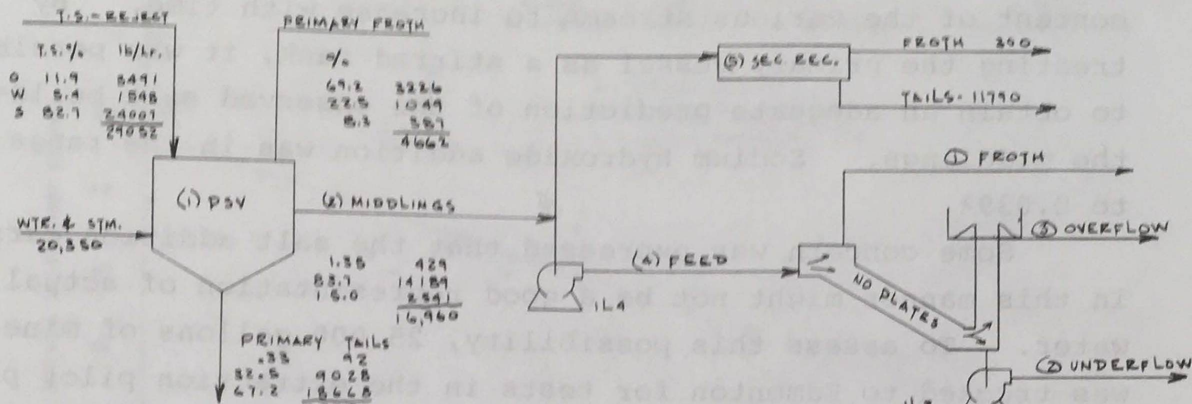
Figures 15 and 16 show that the observed reduction of froth quality with chloride concentration is more or less linear, whereas the reduction in recovery is exponential.

It should be noted that there is no steady state data in the range of 0-1000 ppm chloride. Values obtained by interpolation of the data in this range should not be considered as a totally reliable estimate of the effects of chloride on the process, particularly since the salt test under transient conditions indicated a much greater effect. More work is planned to further quantify these effects.

S.J. Lane

TYPICAL PSV FLOWS & INCLINED SETTLER FLOWS

FIGURE 17



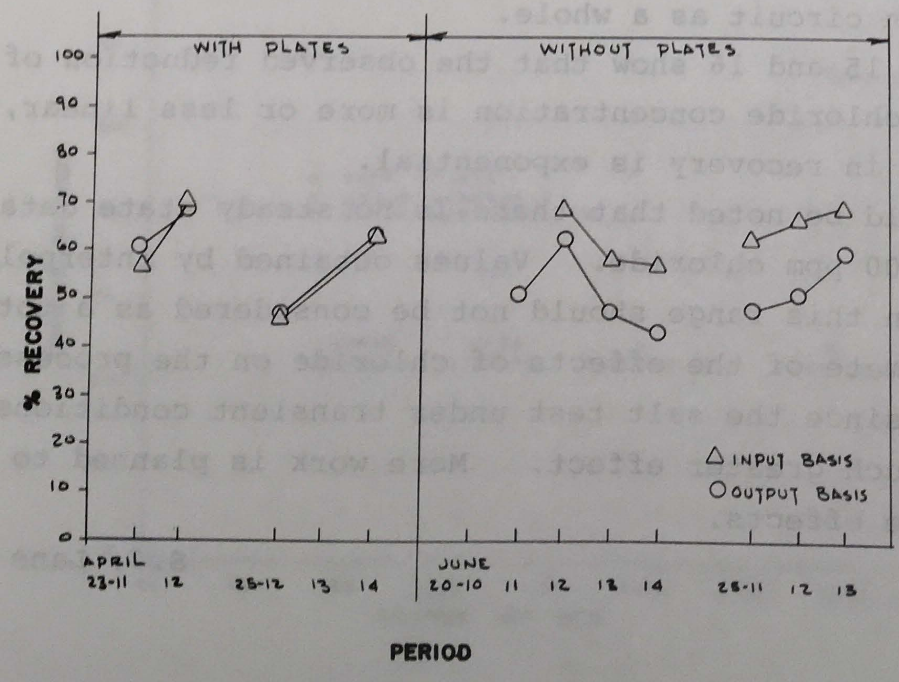
PERIOD		FEED	%	①	%	②	%	③	%
20-11	O	146	3.4	74	60.3	37	1.74	35	1.64
	W	3463	79.6	39	32.2	1553	73.8	1861	88.2
	S	740	17.1	9	7.6	516	24.5	215	10.2
	TOT	4327		122		2106		2110	
20-12	O	59	1.42	37	71.1	11	0.59	11	0.49
	W	3433	82.6	11	20.5	1418	75.2	2004	90.4
	S	664	16.0	4	8.5	456	24.2	204	9.2
	TOT	4154		52		1885		2217	
20-13	O	19	1.10	9	69.8	6	.61	4	.46
	W	1511	83.8	3	20.7	702	76.1	806	93.0
	S	272	15.1	1	9.5	215	23.3	56	6.5
	TOT	1803		13		923		867	
20-14	O	30	1.47	13	65.5	13	1.05	4	.48
	W	696	83.3	6	27.1	979	78.9	712	91.6
	S	311	15.3	1	7.4	248	20.0	62	8.0
	TOTAL	2038		20		1241		777	

PERIOD		FEED	%	①	%	②	%	③	%
25-11	O	42	1.0	20	73.3	12	.62	10	.48
	W	3571	88.7	5	18.2	1590	84.8	1976	93.1
	S	412	10.2	2	8.6	274	14.6	136	6.4
	TOT	4024		27		1875		2122	
25-12	O	27	.9	19	70.8	10	.65	8	.41
	W	3430	84.4	5	19.6	1519	76.8	1906	92.5
	S	596	14.7	3	9.7	447	22.6	146	7.1
	TOT	4066		27		1978		2061	
25-13	O	42	1.0	25	71.0	10	.49	7	.34
	W	3761	87.3	7	19.6	1784	83.3	1970	92.5
	S	503	11.7	3	9.4	347	16.2	153	7.2
	TOT	4307		35		2142		2130	

NOTES: 1) PSV OPERATION IS AVG. OF PERIODS 20-12, 20-14, 25-12
2) MIDDLINGS OBTAINED BY OVERALL BALANCE ON PSV & MEASURED MIDDLINGS COMPOSITION.
3) ALL FLOWS LBS./HR. 4) INCLINED SETTLER FEED CALCULATED FROM OUTPUTS 5) PERIOD 25-12.

INCLINED SETTLER RECOVERIES

FIGURE 18



References

1. *Lane, S.J. "Effect of sodium chloride and saline mine water on the extraction circuit". Extraction Pilot Plant Special Report E-9, June 20, 1974.*
2. *Lane, S.J. "Effect of saline mine water on extraction circuit performance". Extraction Pilot Plant - Special Report E-11, July 11, 1974.*

3.4 Exploratory Extraction

3.4.1 Inclined Settler Studies

Inclined settlers effect gravity separation of components from flow between closely spaced, corrugated, parallel plates inclined at approximately 45°. A series of tests were conducted with a commercial settler to assess the feasibility of processing the middlings stream from the primary separation vessel. The tests included runs to study the effect of loading caused by variations in both feedrate and effective area (1). Effective area was varied by operating with and without the plates.

The configuration of the circuit and typical operating variables are shown by Figure 17 for tests without the plates. Recoveries are plotted in Figure 18. Froth qualities which are not shown are equivalent to those obtained in primary separation. The oil recoveries do not show any systematic dependence on loading, or equivalently, on operation with and without the plates. Consequently, it appears that most of the oil is recovered in the feedwell of the settler. On a volume basis, the recovery is approximately 55% at a feedwell loading of 1314 lb/hr/ft^3. This is significantly larger than expected from the conventional secondary circuit, (Denver cells) which would require twice as much volume to effect the same recovery.

Future Activities

Data obtained by operating the inclined settler in series with a duplicate feed will require analysis and reporting. The available data provide a strong incentive to pursue the possibility of replacing all or a portion of the conventional secondary circuit with inclined settlers or modifications thereof.

F.A. Seyer

Reference

1. *"Inclined Settler in Secondary Extraction". Syncrude Interoffice Correspondence, F.A. Seyer to K.C. Porteous, August 12, 1974.*

3.4.2 Two-Cell Extraction System

The two-cell system, described (1) was demonstrated to be a possible alternative to the present base case extraction system.

When compared with base case runs, using similar tar sand feed, the two-cell unit usually produced satisfactory oil recoveries and froth qualities. The merits of the two-cell operation relative to the normal operating mode of the base case are:

1. Lower temperatures (150 vs. 176°F)
2. 40% less fresh water
3. Elimination of secondary recovery

After minor changes of equipment, froth sampling and weighing techniques, the experimental data showed good reproducibility and consistency.

The experimental program of April 15 through 17, 1974, (2) was undertaken to assess the importance of the sizing of the sand separator underflow orifice and sand separator internals on circuit performance.

Tar sand available for use in these tests was extremely rich (average 13.8% bitumen) and the circuit performed in a steady manner yielding excellent material balances. Recoveries and froth qualities achieved over the entire test period are shown in Table 6.

TABLE 6

	Average	Range
Oil Recovery	96.6	94.7 - 97.4
Froth % Oil	71.9	70.6 - 74.2
% Water	19.0	17.6 - 20.0
% Solids	9.1	8.1 - 10.1

None of the mechanical changes made during the run effected oil recovery. Since the cones within the sand separator are rationalized within the design philosophy of the unit, it is disturbing to find that the circuit performs equally well with and without these internals. It is not known whether this result is generally valid or merely reflects the ease of processibility generally found for rich tar sand.

The quantity of reject material off the tumbler discharge screen during two-cell operation is higher than that experienced by the base case circuit and is the result of a lower slurry temperature. Earlier tests (3) indicated that the amount of reject can be reduced to levels comparable to the base case through the use of high temperature slurry water. Results from the test-work discussed above, tend to confirm this conclusion.

A run in which high fines tar sand was processed in the two-cell system yielded lower oil recoveries but better froth quality than the base case circuit as shown in Table 7.

TABLE 7

Unit	Tar Sand			Combined Froth Quality			Oil Recovery	
	Bitumen %	Fines %	pH	Oil %	Water %	Solids %	Prim	Comb
Base Case	8.6	18.2	8.36	~42.8	~48.6	~8.6	55.6	87.6
Two-Cell	8.17	21.2	7.78	61.3	32.5	6.2	61.6	--

To keep this comparison in perspective, it is important to realize that the base case data are the result of extensive programs aimed at defining suitable operating conditions whereas the two-cell data come from a short experimental program.

At the present time, no additional two-cell work is planned at the 15 TPH scale. The system has certainly demonstrated its mechanical reliability and operability and the basic processing concept warrants additional consideration based on its performance. However, the present design requires a careful review of existing mechanical complexities and reassessment of technical and economic incentives of the concept.

L.M. Cymbalisty
K.C. Porteous

References

1. *Research Department Research Activity, 1973, January, 1974, p. 13.*
2. *"Two-Cell Extraction Tests". Syncrude Interoffice Correspondence, L.M. Cymbalisty to K.C. Porteous, March 25, 1974.*
3. *Research Department Progress Report, September 1, 1973, p. 3.*

FIGURE 19.

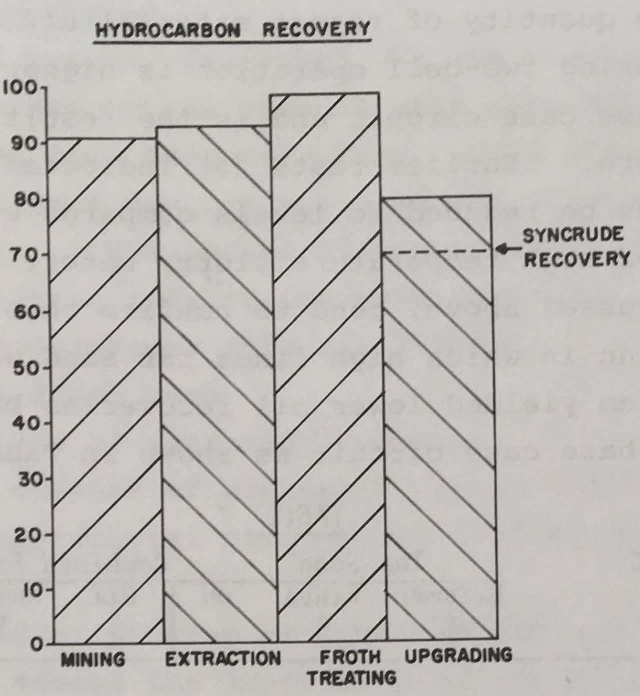


TABLE 8

Overall Plant Energy Balance (total plant flows in TPH)

Inputs	Flow Rate	Enthalpy or Heating Value	Energy 10^9 BTU/hr
1. Bitumen	1183	16750 BTU/lb	39.6
2. Natural Gas	58.5	20496 BTU/lb	2.4
3. Electricity	5×10^6 W-hr/hr	3.41 BTU/hr/W	0.015
4. Water for Tailings	8451	(0 at 40°F)	0.0
5. Cooling Tower	990	(0 at 40°F)	0.0
6. Solids	8567	(0 at 40°F)	0.0
Total			42.0

Outputs			
7. Bitumen Losses			
Reject	12	16750 BTU/lb	0.4
Ext. Tails	71	16750 BTU/lb	2.4
Froth Tails	22	16750 BTU/lb	0.7
8. Coke	101	13600 BTU/lb	2.8
9. Sulphur	42	3000 BTU/lb	0.3
10. Syncrude	780	18250 BTU/lb	28.5
11. Water in Tailings	8451	136 BTU/lb	2.3
Cooling Tower	990	970 BTU/lb	1.9
12. Solids	8567	27 BTU/lb	0.5
13. Stack Gases	2655	128 BTU/lb	0.7
14. Miscellaneous			1.0
Total			42.0

3.4.3 Technology of Hot Water Processing and Research Needs

In order to identify potential research areas and to facilitate rational allotment of priorities a report outlining current hot water processing technology has been prepared (1) Material and energy balances from process design specifications for each area of the plant are the main basis for discussion. The material balance for the upgrading area includes details for some of the components such as sulfur. One of the critical items considered, is the overall water balance of the plant. Water reclaim, and recycle of the reclaim water to extraction, is essential in order to restrict the sludge pond to a manageable size. The long term quality of the recycle water, however, in terms of its effect on extraction, is essentially undefined and consequently there is a large uncertainty in the rate of recycle that can be sustained. Moderate changes in expected recycle (±5% say) when integrated over some 25 years result in an order of magnitude uncertainty in sludge pond volume.

Figure 19 shows the hydrocarbon recoveries expected from each of the plant areas. Overall hydrocarbon recovery of the plant exclusive of mining is the product of 0.93 x 0.98 x 0.79 = 72% of the bitumen feed. The largest potential for increasing recovery is the possibility of utilizing coke produced in the upgrading area. In extraction, advanced technology through research may increase recovery by 7% or less (5% or less for entire plant). Since extraction has been extensively piloted it was concluded that major research effort would be necessary to effect even small increases in extraction recovery. Furthermore, in view of scale-up uncertainty it is not clear that any improvements at the pilot scale can be guaranteed in a commercial operation.

The overall energy balance is a measure of the efficiency of the process and is a useful basis for comparison with alternative processes. Table 8 is an approximate energy balance for the entire process. Net coke and bitumen losses are the largest portions of the available energy which are not recovered. Approximately 68% of the input energy is recovered in the Syncrude product.

Brief consideration is given in the report to other processes. As an example, direct coking of the tar sand is a feasible alternative, to the hot water process. In view of successful pilot scale tests of the direct coking of tar sand the probability of developing a process at a commercial scale is high.

The large incentives for direct coking are a result of complete elimination of the extraction and froth treating steps and associated major problems with water usage. One of the main questions concerning feasibility of the coking process is the energy loss resulting from reject of high temperature solids at about 1400°F (1) Table 9 gives a comparison of that portion of the energy losses attributable to the hot water process as compared to direct coking.

TABLE 9: COMPARISON OF ENERGY LOSSES

Energy Lost Because of:	Hot Water Process	Direct Coking
bitumen lost in extraction and froth treating	172 BTU/lb T.S.	0 BTU/lb T.S.
hot extraction water to ponds	113 BTU/lb T.S.	0 BTU/lb T.S.
solids to mine	23 BTU/lb T.S.	230 BTU/lb T.S.
Total	308 BTU/lb T.S.	230 BTU/lb T.S.

The table shows that even with solids reject at 1400°F direct coking has a much higher thermal efficiency than the hot water process. Energy lost because of bitumen losses from extraction and froth treating are almost equivalent to the energy necessary to heat the solids. Furthermore, making the comparison even more dramatic, there is no doubt that a substantial portion of the high grade energy of the solids at 1400°F can be recovered economically, while the low grade energy in extraction tails is essentially unrecoverable.

F.A. Seyer

References

1. Seyer, F.A., "Technology of Hot Water Processing and Research Needs - Some Observations," 48 pp, September, 1974.
2. "A preliminary comparison of direct coking of tar sand vs. hot water extraction with fluid coking of bitumen". Syncrude Interoffice Correspondence, Ref. No. 22601, P.C. Flynn to R.R. Goforth, et al, Dec. 5, 1974.

3.4.4 Coalescence Study

Microscopic examination of froth samples taken from the middlings froth interface suggest that young froth is a mass of particles squeezed together in a "bunch of grapes" mode. This structure favours the entrapment of solids laden water and it is resistant to chemical or physical treatment aimed at improving froth quality. A study was undertaken to gain a better understanding of the factors affecting coalescence of bitumen drops. Fast coalescence, that is, the action of two or more drops coming together and forming one drop, is desirable for good quality froth, however, excessive coalescence before the froth formation stage may lead to large bitumen losses and/or dirty froth.

The bitumen-water system is not suitable for direct observation, consequently a series of model studies were undertaken using paraffin oil or paraffin/toluene mixtures in water with and without contaminants.

To simplify the system further it was decided to observe the behavior of a drop of oil rising through the water phase to the surface on which the same oil phase was present. Thus, any effect of curvature was present only in one drop and one coalescing surface was flat. The driving force for coalescence includes components of cohesion (surface tension) and buoyancy.

Some of the results, reported in full in references (1,2) were unexpected.

1. Effect of pH. Values of pH of 4 and 9 were most favorable to rapid coalescence, whereas those approaching 10 were unfavorable.
2. Effect of Potassium Chloride. Using 10^{-3} molar potassium chloride in the aqueous phase showed that this electrolyte impedes coalescence at low pH, but that it reduces coalescence time appreciably in the alkaline range.
3. Effect of Electric Field. Grounding of the interface reduces coalescence time and eliminates the slow change of coalescence time with the age of the interface.

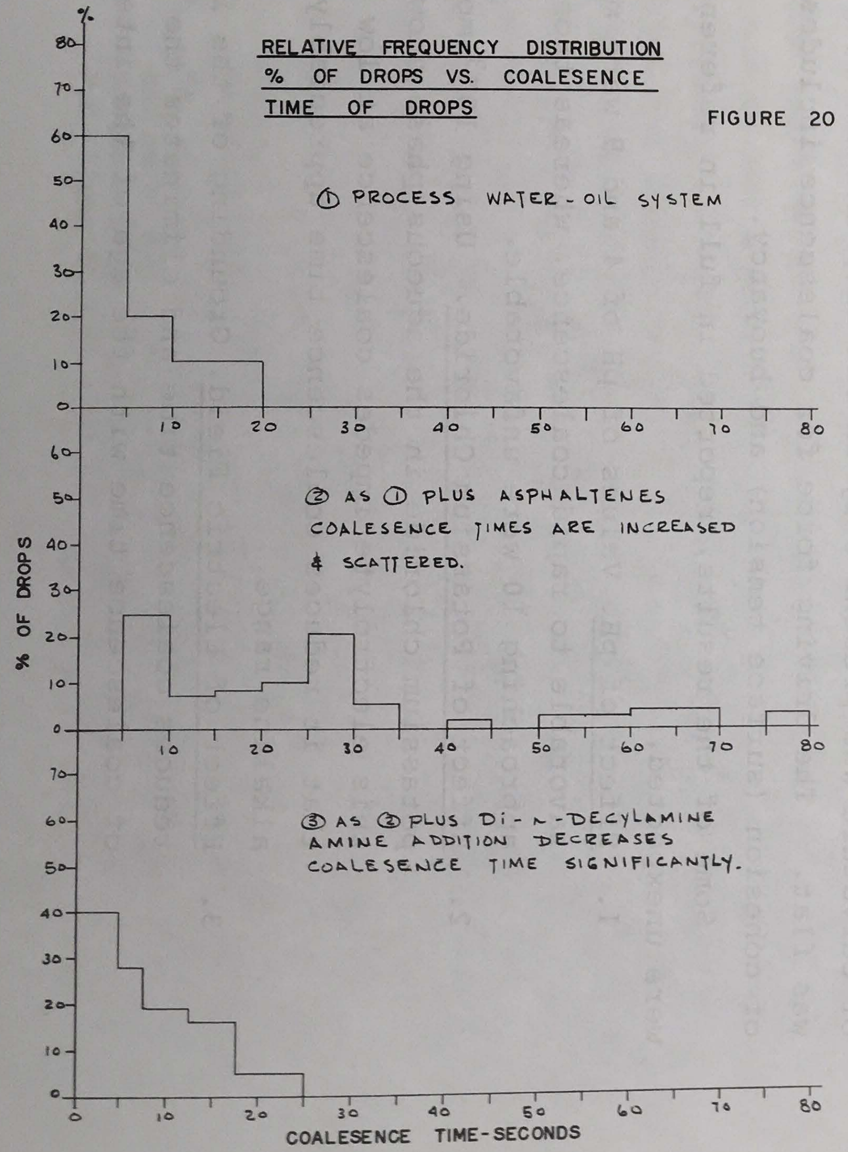


FIGURE 20

RELATIVE FREQUENCY DISTRIBUTION % OF DROPS VS. COALESENCE TIME OF DROPS

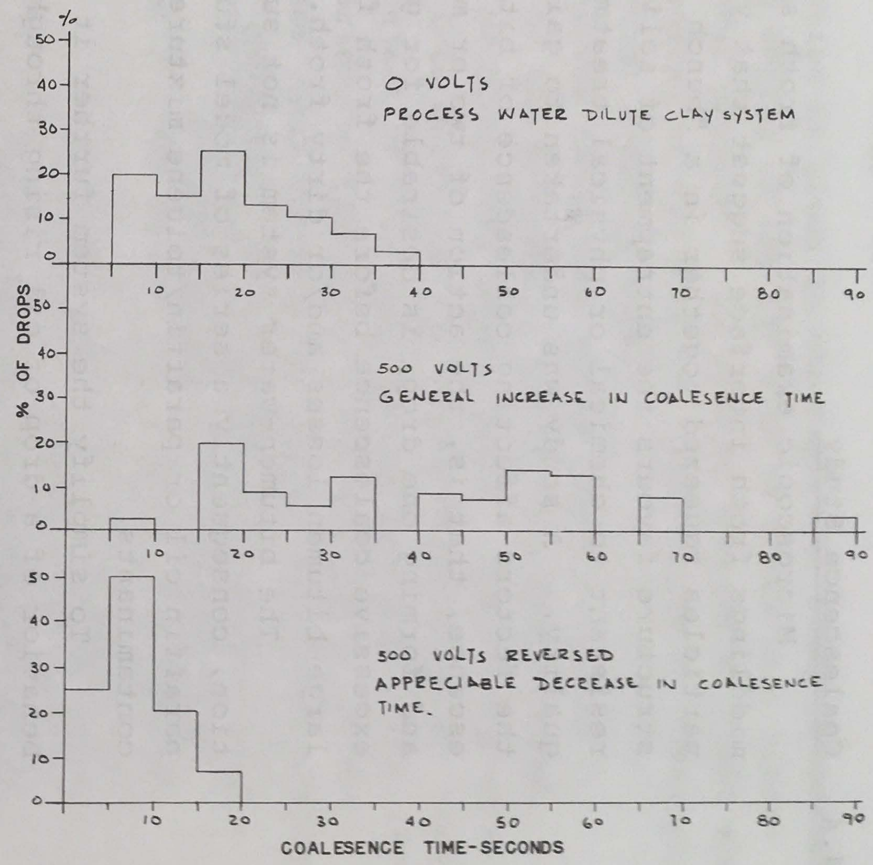


FIGURE 21.

RELATIVE FREQUENCY DISTRIBUTION % OF DROPS VS. COALESENCE TIME OF DROPS

4. Effect of Bitumen. A series of experiments were carried out in which bitumen was dissolved in the toluene/paraffin mixture. With this system the following results were obtained:
   a) Coalescence times were not affected by the presence of bitumen in the toluene/paraffin droplets.
   b) Asphaltenes dispersed in the water phase considerably increased coalescence time.
   c) Carbon dioxide addition caused a slight decrease in coalescence time.
   d) Clay, when added to the system in a small amount, increased coalescence time, but further additions caused a marked reduction.
   e) Amines significantly reduce coalescence time (see Figure 20).
   f) Application of 500 V DC between the oil and water sides of the interface increased or decreased coalescence time according to the polarity of the electrodes. (See Figure 21).

It is concluded that certain additives (amines, clays) will favorably affect coalescence times, so will certain physical forces (electric fields).

It is not yet possible to incorporate these findings in an extraction scheme since these agents are also known to adversely affect recoveries. Maybe the mechanism, by which they reduce recoveries, is through increased premature coalescence which would cause large bitumen globs to form. When these are solids laden, recoveries would certainly drop. This raises the question as to whether or not asphaltenes have a beneficial effect on recoveries through an increase in coalescence time, thus maintaining individual bitumen drops or whether they promote aeration through the provision of hydrophobic surfaces (3).

E.D. Cooke
R. Schutte

References

1. *Research Department Progress Report, September 1, 1974, p. 5.*
2. *Research Department Progress Report, October 1, 1974, p. 4.*
3. *Research Department Progress Report, May 1, 1974, p. 13.*

3.4.5 Bitumen Composition and Flotation

Disproportionation of tar sand bitumen during hot water processing was reported on in the 1973 report (1). The studies were continued in 1974 to include secondary recovery and the relationship between froth quality and bitumen composition.

A workup of the data collected from the 15 TPH run on average tar sand showed that the extraction process is most efficient for asphaltenes, followed by resins, saturates, and aromatics. Recoveries for individual components were calculated on an output basis:

Overall oil recovery	91.4%
Asphaltenes	92.4%
Resins	91.7%
Saturates	90.3%
Aromatics	90.7%

This study and the results given previously (1), led to the postulate that the presence of asphaltenes (colloidally dispersed solid hydrocarbons) is essential to air attachment. Hence, a study was undertaken on the effect of asphaltenes as additive to the process. These experiments are reported on in Section 3.4.7.

Several hypotheses are capable of explaining the observed yield pattern. It is plausible that light hydrocarbons such as saturates and aromatics, are leached by the process water, may be helped by some of the resins, especially resins II. A precedent for this theory is set by the familiar caffeine number determination where caffeine facilitates an enhanced solubility of large aromatic molecules in water. Finally, it is known from earlier studies, references (2,3), that the gas phase in the froth is depleted in oxygen within a short time (24 hours). Bitumen components may be readily oxidized in the sequence: saturates and aromatics $\longrightarrow$ resins $\longrightarrow$ asphaltenes. The observed enrichment in asphaltenes in the froth could reflect the oxidative processes. However, these reactions fail to explain the low polar content of tailings bitumen.

1. Secondary Recovery

Three series of secondary froth produced by a Maxwell cell and six Denver cells all in series, were analyzed; detailed results are reported in reference (4). The results were inconclusive since certain trends were observed in only two of the three series. A complicating factor was the tar sand used: two series with high fines and one with average tar sand.

One conclusion may be drawn: the oil recovered in each cell differs from the oil recovered in the preceding cell and from that recovered in the following cell. Any in-depth study of secondary-circuit performance should treat each cell individually since air attachment is a function of oil composition.

2. Froth Quality

To enable a study of froth quality as a function of bitumen composition, a method was devised to enable the gathering of a large number of data in a relatively short time. It had been observed while studying spectroscopic methods of bitumen analysis (see Section 5.2), that the absorbance in the visible region of the spectrum was not constant. Data were gathered on this variability and on froth quality. Not only does the absorbance of bitumen vary over a wide range; from 0.2 to 0.3 A l/g (see Table 10 for an example), there was also a significant correlation between absorbance at 600 nm and the water content of the froth, see Figure 22.

Several mechanisms by which this may happen have been proposed and are described in reference (5). It seems clear though that froth quality is a function of bitumen composition. This adds a new dimension to the optimization of the extraction circuit and it may explain a large part of the unpredictable relationships of froth quality as to conditions.

The variability of the absorbance at 600 nm indicates that spectroscopic methods for the determination of bitumen quantities must be used with caution, there is no standard bitumen.

Further studies are planned into the nature of the colour centres, with the objective of forcing each bitumen type to become standard as far as colour is concerned, for instance through the addition of compounds which readily form charge transfer complexes.

R. Schutte

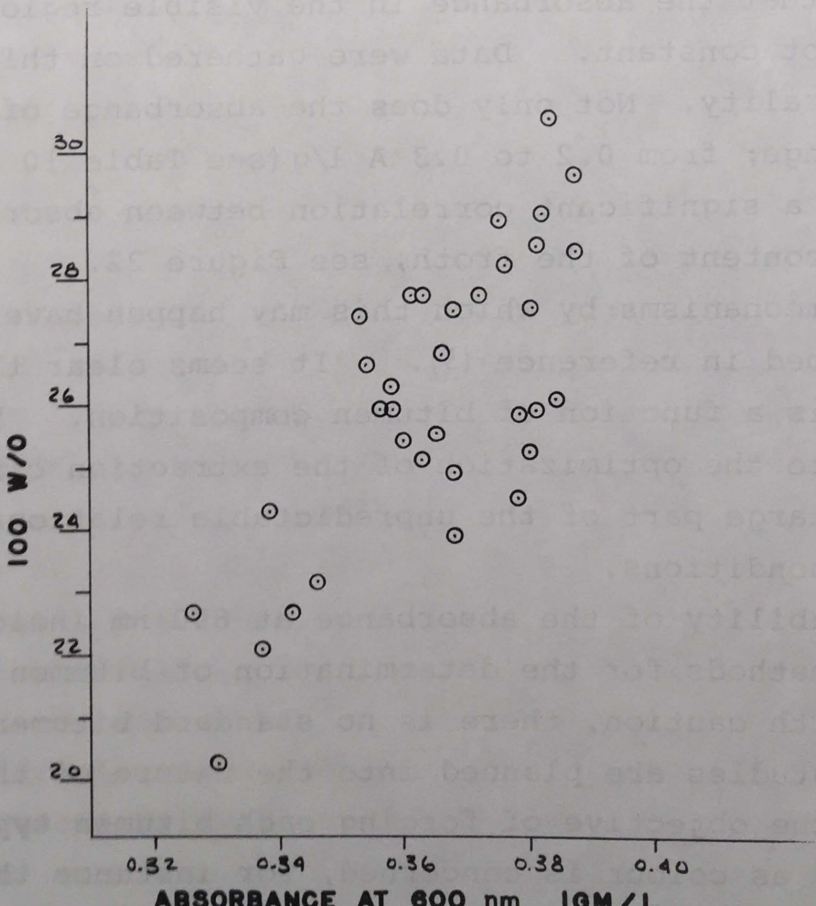
TABLE 10

Absorbance at 600 nms. of Bitumens from Two-Cell Extraction Streams

Stream	No. of Samples	Average Absorbance	Absorbance Range
Tar Sand	23	0.361	0.342-0.385
Reject	23	0.270	0.186-0.367
Flooded Slurry	24	0.356	0.328-0.384
Froth	93	0.360	0.323-0.389
Middlings	21	0.211	0.058-0.268
Tailings	129	0.228	0.095-0.358

FIGURE 22.

TWO-CELL EXTRACTION FROTH



References

1. *Research Department Research Activity, 1973, January, 1974, p.32.*
2. *Research Department Research Report, 1967, Syncrude Canada Ltd., Report S-2M-67, p. 59.*
3. *Research Department Research Report, 1966, Syncrude Canada Ltd., Report S-1M-66, p. 53.*
4. *Research Department Monthly Progress Report, March 1, 1974, p. 21.*
5. *Research Department Monthly Progress Report, June 1, 1974, p. 7*

3.4.6 Characterization of Bitumen from a High Oil - Low Fines and a Low Oil - High Fines Tar Sands Extracted Under Various Conditions of Chemical Addition

Addition of sodium chloride at the primary separation stage is normally detrimental to the hot water process, resulting in serious oil losses. (1,2) It has been demonstrated, however, that salt addition can lead to improved oil recovery for certain types of tar sand (2). Likewise, although sodium hydroxide is normally regarded as an improver for the hot water process, addition of excess reagent may lead to decreased oil recovery. (2)

As part of a continuing program to increase understanding of salt/alkali effects, hydrocarbon-type analyses (3,4) were carried out on bitumens extracted from various process streams. These were derived from SS Pot experiments in which the above reagents were added.

In summary the results were:

1. The asphaltene content of non-recovered oil (middlings and tailings: was lower than that of floated oil.
2. Increased primary oil recovery was associated with increased resin I and decreased aromatic content of the tailings.
3. In the case of the low oil tar sand, aromatics were enriched in the water phase.

The main conclusion of this work is that, although selectivity of certain bitumen fractions is usually achieved during the hot water process, (for example, oil from middlings, and tailings streams tend to be low in asphaltenes) some of this selectivity is lost in the presence of added chloride ion when that deteriorates tar sand processibility.

Future Activities

Electrophoretic and light scattering study of the bitumen components in the various streams where the processibility drastically changes, will be undertaken to understand the charge, size and shape of the particles in their environments.

It is possible that the high aromatic content of the water phase is due to water soluble compounds. This presents an oil contamination problem of a rather different nature.

Further examination will be made and the effects of dissolved oil on sludge settling will be studied.

J.E. Filby
M.V. Baptista

References

1. *Research Department Progress Report, August 1, 1974, p. 8.*
2. *Research Department Progress Report, February 1, 1974, p. 11.*
3. *Research Department Progress Report, July 1, 1974, p. 15.*
4. *Research Department Progress Report, October 1, 1974, p. 11.*

.7 Tar Sand Processibility Aids

The problem of disposal of saline water from mine dewatering operations has been the subject of much recent discussion (1). Although the possibility of pumping this water to the tailings pond has been considered, recent pilot plant work demonstrated that the presence of chloride ion in process water has a particularly damaging effect on the extraction process (2). This is reviewed in Section 3.3.

Although other options exist for the disposal of mine water and analyses of core samples (particularly those from the N.W. corner of the mining area) indicate that some tar sand deposits contain sodium chloride at levels as high as 2500 ppm of chloride (3), reference is made to Section 2.1. It now seems certain that sodium chloride will eventually enter the extraction circuit, regardless of which method is chosen for the disposal of mine water.

Since certain chemicals, notably humic acid derivatives (4) and asphaltene derivatives (5), have been proposed as flotation aids for the primary extraction process, it was of interest, therefore, to test these chemicals in the presence of sodium chloride.

Since the production of humic acid species is simple (6) these chemicals were also tested to evaluate their effect on the processibility of a low-oil tar sands and to investigate if they can inactivate the harmful effect of salt water in the processibility of tar sands (see Section 3.3).

The disadvantages of using a batch-type apparatus such as the laboratory 500 g extraction unit, to model behaviour in the extraction circuit, have been noted. It was thought that by careful control of conditions meaningful results could, however, be obtained (7).

A large number of results have now been accumulated using the laboratory 500 g extraction unit. These are reviewed, for convenience, under the headings:

1. Addition of Asphaltene related substances, and
2. Effect of humic species.

1. Asphaltene Derivatives

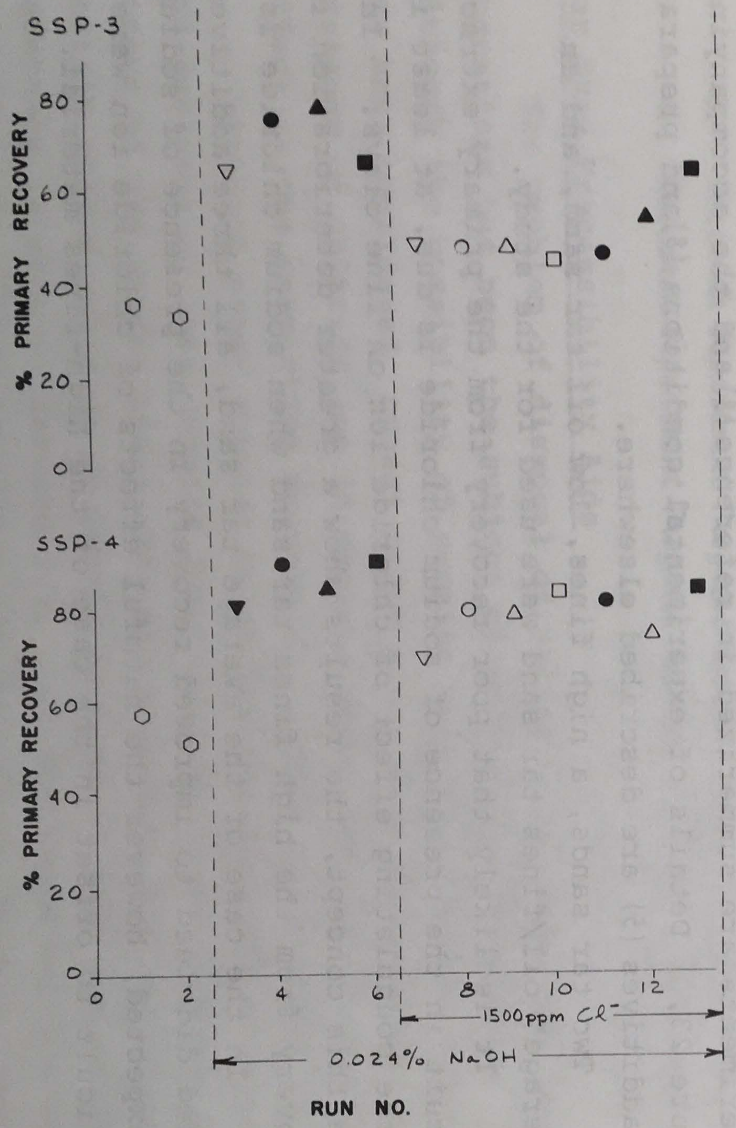
The results from two series of 500 g batch extraction experiments are summarized in reference (8) and the accompanying Figure 23, Details of experimental conditions (8) and preparations of additives (5) are described elsewhere.

Two tar sands, a high fines, low oil tar sand, and an "average" oil/fines tar sand were used for the study.

It is likely that poor recovery from the primary extraction circuit in the presence of sodium chloride is due, at least in part, to the coagulating effect of chloride ion on fine clays. In keeping with this concept, the results show a greater deterioration in recovery from the high fines tar sand when sodium chloride is present.

In the case of the average tar sand, all three additives tested did lead to improved recovery in the presence of sodium chloride. As expected, however the harmful effects of chloride ion were more difficult to offset in the case of the high-fines material.

FIGURE 23.



KEY TO SYMBOLS

⬡ - BLANK

▽ - NaOH BLANK

● - 0.16 % ASPHALTENES

▲ - 0.16 % OXIDIZED ASPHALTENES

■ - 0.16% SYNTHETIC HUMIC ACIDS

○ - 0.02 % ASPHALTENES

△ - 0.02 % OXIDIZED ASPHALTENES

□ - 0.02% SYNTHETIC HUMIC ACIDS

ANALYSIS OF TAR SANDS

SAMPLE	P.S.D. %(-325)	% OIL	% WATER	% SOLIDS
SSP-3	19.94	8.04	6.29	85.67
SSP-4	12.89	9.18	7.39	83.43

NOTE: ALL CHEMICALS ARE REPORTED AS WT. % ON TAR SAND BASIS WITH THE EXCEPTION OF SODIUM CHLORIDE. THIS IS REPORTED AS ppm Cl^- ON TOTAL WATER BASIS.

Recent experiments have demonstrated that oxidation of pentane asphaltenes leads to humic-acid like substances which behave as dispersing agents.

It seems probable that, by the addition of suitable dispersing agents, the primary extraction process might be made operable, even in the presence of relatively high chloride ion concentrations.

A more attractive approach would be to generate dispersing agents in situ, either in the tar sand or at the slurry stage; it might turn out, for example, that oxidation (as opposed to drying-out) of tar sand is beneficial to the extraction process.

Moschopedis and Speight, at Alberta Research have devoted considerable attention to the oxidation of tar sand with a view to generating emulsifying agents suitable for in situ extraction schemes. These workers succeeded in achieving mild oxidations using a dilute stream of ozone in air (9).

J.E. Filby

2. Effect of Humic Species

Humic acid in the sodium humate form (0.020 wt %) promotes better recovery than sodium hydroxide alone (10). When added in conjunction with sodium chloride (500 ppm chloride on a total water basis) in the slurry the recovery is drastically reduced (10). This is probably due to i) a decrease in humic acid charge by the sodium chloride making the humic acid aggregate (6), and ii) the complexing of humic acid with Ca^{++} ions released from the system by the sodium chloride (6). If there is no humic species available for the cation exchange with clays, dispersion of these is not possible (1). Higher levels of humic acids should be added.

When humate is added to the slurry and sodium chloride in the flooding step, the recovery improves over that found when they are added together in the slurry (10). This is assumed to be due to the fact that the humate promotes ion exchange with the clays before the sodium chloride addition.

Lower levels of humate species addition (0.002 to 0.011 wt %) do not promote any benefit at the same dosage of sodium chloride. Similarly higher concentrations of salt (1500 ppm chloride) were also more harmful *(11)*.

The use of sodium salts of sulfomethylated humic acids (0.020 wt %) appears to correct the harmful effect of sodium chloride (1500 ppm chloride) by improving the oil recovery and drying the froth *(12)*.

Future Activities

Continuation of the study of chemical addition of humic acid in a large range of humic concentration, ionic strength and metal-ion activity is planned.

The humic species could be prepared from waste materials already existing at the tar sand site and from other wastes simulating those produced in the plant in operation (coke).

M.V. Baptista

References

1. *Research Department Progress Report, June 1, 1974, p. 36.*
2. *Lane, S.J. "Effect of saline mine water on extraction circuit performance". Extraction Pilot Plant - Special Report E-11. July 11, 1974.*
3. *Research Department Progress Report, October 1, 1974, p. 40.*
4. *Research Department Research Activity, 1973, p. 19.*
5. *Research Department Progress Report, August 1, 1974, p. 1.*
6. *Research Department Progress Report, December 1, 1974, p. 12.*
7. *Research Department Progress Report, September 1,1974, p. 1.*
8. *Research Department Progress Report, November 1, 1974, p. 1.*
9. *Moschopedis, S.E. and Speight, J.G. "Oxidation of bitumen in relation to its recovery from tar sand formations". Fuel, 53, (1) 21-25 (1974).*
10. *Research Department Progress Report, August 1, 1974, p. 1, Table 1.*
11. *Research Department Progress Report, August 1, 1974, p. 1, Table 2.*
12. *Research Department Progress Report, November 1, 1974, p. 3, Table 1.*

3.4.8 Non-Aqueous Extraction of Bitumen

The extraction of bitumen directly from tar sand with a light naphtha (b.p.<100°C) has several desirable features:

1. The ease of the extraction operation, especially settling time and temperature (<60°C).
2. The quantity (up to 90% of total) and quality (virtually solids, water free) of bitumen obtained.

A limited amount of work has been carried out (1) to date to support the above remarks, however, there are, of course a number of problems associated with this approach. Not the least of which are the potential loss of solvent and the need to contain light hydrocarbon vapours. However, the following potentials are believed to justify further studies:

a) Extracting tar sand with a light naphtha to yield a bitumen having desirable properties. The residue is either burned to supply energy or extracted with a suitable solvent such as benzene to recover some flotation promoters (asphaltic and humic materials). In this case may be 20% of the tar sand feed to a plant is treated in this fashion.

b) A non-aqueous prewash step of tar sand prior to the hot water process is performed. Such a step removes a major portion of the air-flotation supressors (light hydrocarbons), it wets the remainder of the bitumen (decreases its density) which may render it more amenable to flotation. A possible result of this will be that the hot water process may be conducted at less severe conditions (temperature, and mixing) such that the disintegration of clay lumps is largely avoided, thus alleviating the sludge storage volume required.

L.H. Ali
R. Schutte

Reference

1. *Research Department Progress Report, December 1, 1974, p. 10.*

3.4.9 Laboratory Scale Extraction Unit

There are, to date, two experimental units available for study of the physicochemical interactions of the hot water process. One, a large scale unit (15 TPH to be supplemented by a scaled down version at 2.5 TPH nearing completion, see Section 10.1), and the other a small batch unit with a capacity of 500 g tar sand.

Neither of these units are satisfactory. The problem lies in the large amount of water used in a batch mode in the 500 g unit, on the one side, and on the other in the large scale of the 15 and 2.5 TPH units. Their scale precludes the use of chemicals that may be available, in the research stage, in quantities of only a few hundred grams, or the use of limited amount of tar sand samples.

Studies are planned of such physicochemical phenomena as:

1. Solubility of hydrocarbons in process water as a function of process water quality (sodium chloride) see Section 3.4.6). This information is needed to determine the toxicity and the suitability for recycle of the pond water.
2. The influence of organic chemicals on clay dispersion (Section 3.4.7), coagulation (Section 3.4.4), sludge compaction (Section 3.6) and froth quality (Section 3.4.5).
3. The causes of the wide variations in response to the hot water process for different kinds of tar sands.

To fill the gap and to make meaningful samples available for research purposes, preliminary studies were made on the design of a 10 kg/hr extraction unit (1,2,3) that will, as currently envisioned, be able to process that quantity on a continuous basis.

L.H. Ali
R. Schutte

References

1. Research Department Progress Report, September 1, 1974, p. 9.
2. Research Department Progress Report, December 1, 1974, p. 9.
3. "Small Laboratory Extraction Circuit (SLEC)". Syncrude Interoffice Correspondence, L.M. Cymbalisty to L.H. Ali, November 21, 1974.

3.5 Colloid Studies

In the various streams of the extraction process, stable suspensions are in some cases desirable and in other cases undesirable (1). It is therefore important that we know how to stabilize and de-stabilize colloids.

A comprehensive review of the colloid literature related to tar sand processing was therefore done and the mechanisms of colloid de-stabilization studied in detail (1).

A program was proposed, using a variety of techniques such as light scattering, electrophoresis, charge determination, electron microscopy, infrared and UV spectroscopy, and analytical tools for ionic and mineral concentration determination, to elucidate the boundaries of the so-called "stability domains" (2). They consist of graphical presentations of various solubility and stability regions as a function of two parameters, e.g., pH and electrolyte concentration, keeping all other conditions constant.

Light scattering measurements will be carried out with the Brice-Phoenix spectrophotometer, recently acquired. The turbidity of colloidal suspensions indicates the changes in the nature of the dispersed systems. The transition boundaries between areas of different properties are determined from the abrupt changes in turbidity as a function of the pH or concentration of added chemical. Light scattering is measured on supernatant solutions. Thus, very low or zero turbidities indicate either systems in which precipitate did not form, on systems with settled flocks, leaving the supernatant solutions clear. The corresponding settling volumes help in distinguishing between the two possible situations.

Once the domain boundaries are established, the surface charge characteristics can be determined by electrophoresis. The zero point of charge can be obtained as a function of pH.

For the time being, only electrophoresis mobilities and zeta potentials (assuming dielectric constant and viscosity are the same as for pure water) have been measured on the colloidal particles of the water phase of centrifuged middlings when sodium chloride and hydroxide are used as additivies in the hot water processing of a low oil-high fines and a high oil-low fines tar sands. The processibility of these tar sands is described in reference (3).

FIGURE 24.

EFFECT OF SODIUM CHLORIDE & SODIUM HYDROXIDE ADDED TO EXTRACTION ON THE ELECTROPHORETIC MOBILITY & ZETA POTENTIAL OF WATER PHASE FROM CENTRIFUGED MIDDLINGS

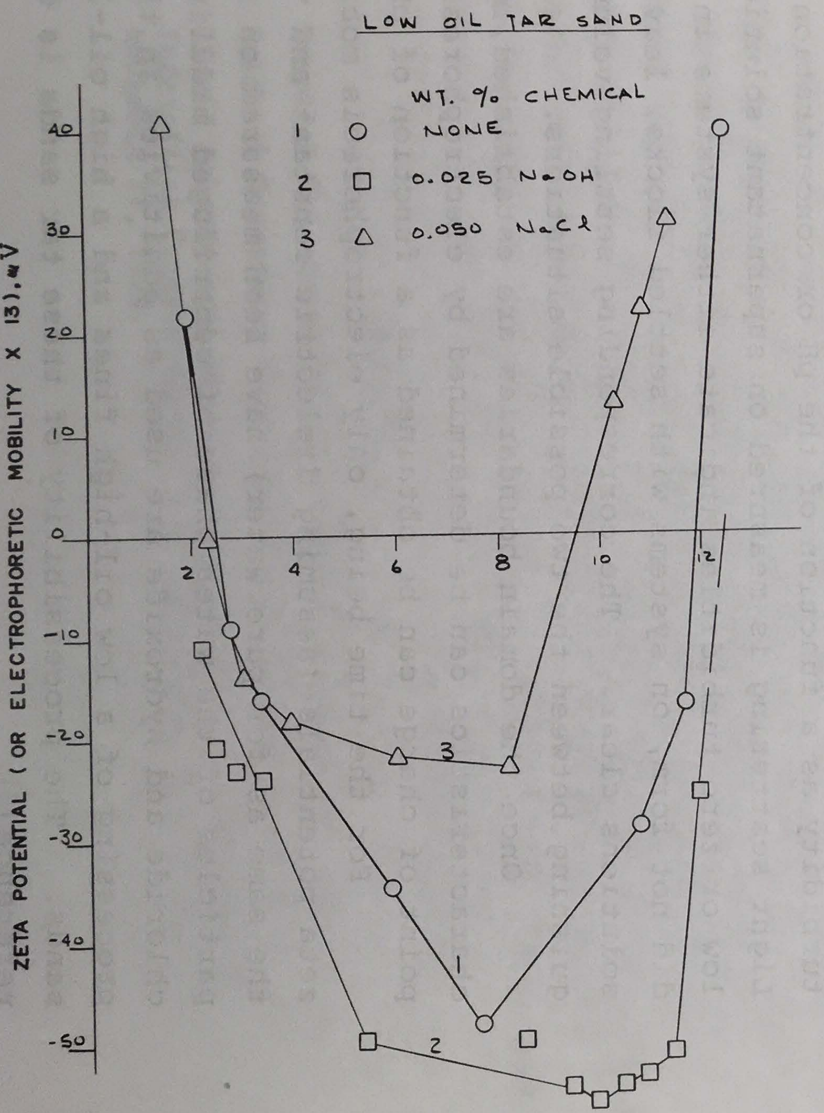
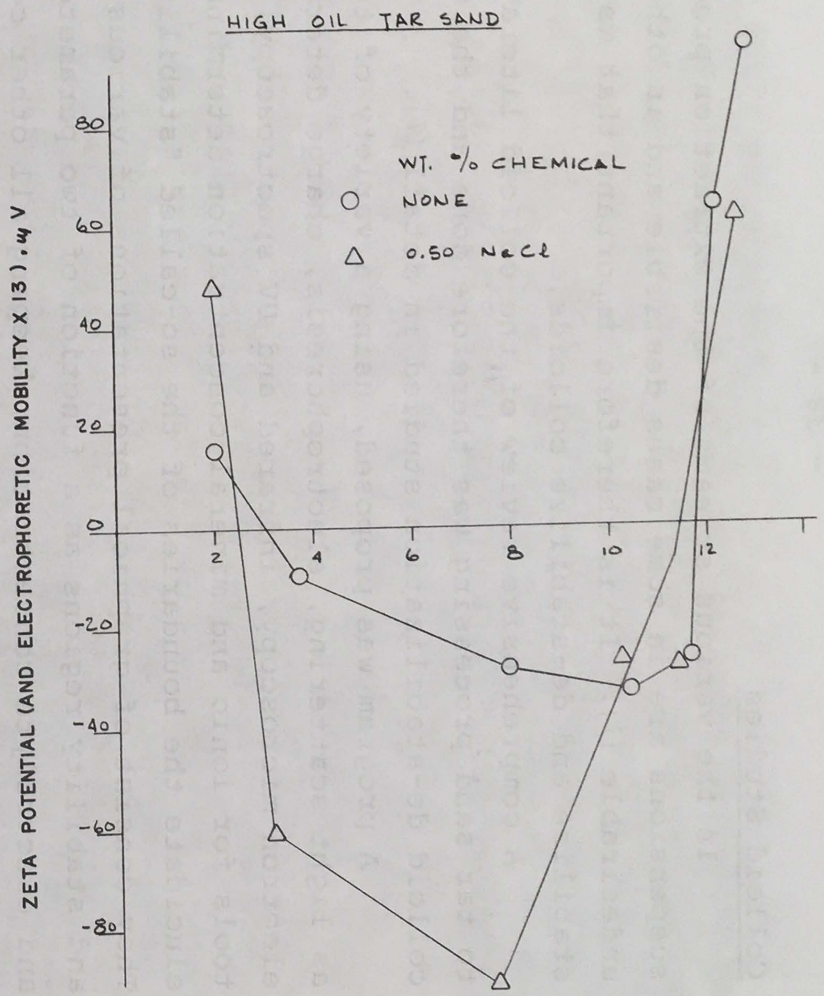


FIGURE 25.

EFFECT OF SODIUM CHLORIDE ADDED TO EXTRACTION, ON THE ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL OF WATER PHASE FROM CENTRIFUGED MIDDLINGS



The main results achieved are:

1. <u>Low oil tar sand (4), Figure 24</u>
   In the presence of sodium chloride (which had a bad effect on the processibility) (3), the electrophoretic mobility and the zeta potential, which were negative, decreased in absolute value, the isoelectric point in alkaline medium decreased from pH 12 to pH 9.5 (3). Curves 1 and 2 appear to represent silica. Curve 3 indicates coagulation of modified silica surfaces (3,4). The decrease in electrophoretic mobility by sodium chloride is an indication that silica particles are aggregated. Thus, the decrease in processibility may be due to the coagulation of silica particles by sodium chloride. It is impossible to state the exact cause without studying the effect of sodium chloride on the electrophoretic mobility of the oil particles.
2. <u>High oil tar sand (3), Figure 25</u>
   Sodium chloride increases the negative zeta potential of the the solids (3). Better dispersion is therefore expected. In fact, sodium chloride increased the oil recovery (3). The shape of both curves indicate the presence of silica (3,4).

M.V. Baptista

References

1. *Research Department Progress Report, June 1, 1974, p. 9.*
2. *Research Department Progress Report, June 1, 1974, p.12.*
3. *Research Department Progress Report, June 1, 1974, p.15.*
4. *Laskowski, J. and Kitchener, J.A. "The hydrophilic-hydrophobic transition on silica". Colloid Interface Sci. 29 (4) 670-679 (1969).*

3.6 Sludge Compaction by Freezing

It is known from literature (1,2) that during the freezing of top layers of certain suspensions. salts migrate to the unfrozen dispersion. The increase in the metal-ion content in the bottom layers may promote coagulation of the particles. The degree of salt migration depends on the nature of the suspension (3). Organic impurities and acids vary in their migrational behavior (1,2).

Total freezing and subsequent thawing of tar sand effluent streams have already been shown to agglomerate suspended solids (4,5).

To investigate the feasibility of densifying sludge by freezing the upper layers of the suspensions in ponds, and to study the pumpability of the sludge, a program was initiated using two different suspensions from the 15 TPH unit, both extracted with sodium hydroxide (6). Ninety-seven percent of the solids in these suspensions were smaller than 30 micrometers. The partial freezing of these samples was carried out in plastic containers well insulated at the bottom and sides with fiberglass and polyurethane foam. Freezing rate, solids concentration, percentage of volume frozen and chemical addition were the parameters studied.

The main results of the program were. (6)

1. Partial freezing (from 20 to 70%) of the suspensions (8.3 and 6.7% solids) from the top to the bottom, resulted in a sludge with a density of the same order as that obtained when the suspension is settled for the same period of time (41 to 52 wt % solids). The solids concentrations of the ice layers after thawing, varied from 1.8 to 2.7% which are higher than those obtained by total freezing and thawing of previous samples extracted without sodium hydroxide (4).
   Also, total freezing followed by thawing of suspensions that had been extracted with sodium hydroxide, in which 25 and 75% of the top layers were discarded, resulted in almost clear water above the sludge.
2. Varying the freezing rate did not change the density of the sludge.

3. When sludges were remixed with their unfrozen dispersions and allowed to settle, the final densities were, in general, higher than those of the original sludges. This suggests that transportation of sludge with its medium does not decrease the density. Hence, pumping is not likely to have an adverse effect on the densification process.
4. Sludges obtained by freezing 50 and 60% of top layers could be removed with a Moyno pump at the temperature of 1°C. The transportation was, however, easier when the sludge was mixed with the residual dispersion in contact with it. In the latter case, the sludge density after settling of the transported mixture was not severely affected by the transport.
5. Some chemicals such as Alchem 8863, alum, aluminum sulfate, sulfuric acid, and sodium chloride did not have any effect in increasing the density of the sludge, nor in the clarification of the upper layers, when added to the suspensions before partial freezing.

Future Activities

When secondary tailings are allowed to settle, various strata are formed. The effect of freezing of these layers on the agglomeration of solids will be investigated.

Addition of chemicals will await the results obtained from colloid stability (Section 3.5).

M.V. Baptista

References

1. *Research Department Progress Report, March 1, 1974, p. 20.*
2. *Applied Science Laboratories Inc. "Purification of Mine Water by Freezing" U.S. Environmental Protection Agency. Water Pollution Control Research Series 14010, DR202-71, 1971, p.1.*
3. *Rose, A. and Sweeney, R.F. "Waste Water Renovation, Part 2, Feasibility Tests of Freezing" Public Health Publication No. 999-WP-4. Cincinnati, Ohio, 1963.*
4. *Syncrude Internal Report No. 32, August, 1972, p. 6.*
5. *Elliott, O.M. "Freeze-thaw Separation of Solids from Tar Sand Extraction Effluents" U.S. Patent 3,751,358, August 7, 1973.*
6. *Research Department Progress Report, April 1, 1974, p.10.*

4. FROTH AND FROTH TREATMENT

4. FROTH AND FROTH TREATMENT

4.1 Dilution Centrifuging

4.1.1 Dilution Centrifuging Pilot Plant

Activities described in last year's research report (1) spanned the time period from startup through October 1973, which concluded the formal machine evaluation programs for the Westfalia, Merco and DeLaval disc centrifuges. During November 1973, a special centrifuge designed by Bird Machine Company for the one stage separation of water, solids and hydrocarbon was installed and tested in the pilot plant under the supervision of Bird personnel. ARCO have specifically asked to be excluded from discussions relating to the equipment or tests. Therefore, no further information is contained herein and complete details are available on request.

During the period December 1973, through April 1974, the pilot plant ran fairly regularly in order to complete the series of special programs outlined in (2). Based on the performance of the various disc centrifuges in the pilot plant, it appeared that DeLaval was the preferred supplier for the commercial plant and as a result the DeLaval SX 204-T was the disc machine used for these tests. Figures 26 and 27 are process flow diagrams for the scroll and disc centrifuge portions of the pilot plant.

From an operating point of view, the centrifuges themselves presented few real problems. Nozzles in the disc machine tended to plug during the processing of froth from high fines tar sand but this difficulty was eliminated by using larger nozzles in the centrifuge. The naphtha recovery portion of the circuit was troubled by the cold weather which caused a number of lines to freeze. Several things were done to remedy this situation. Boilers were operated 24 hours a day, 7 days a week to provide steam which was used to prevent freezing in certain lines and vessels. Other lines were left filled with naphtha during shutdowns. Frozen instrument air lines caused the fired heater in the naphtha recovery circuit to overheat and resulted in coked heater coils. De-coking involved the use of diesel fuel under

PROCESS FLOW DIAGRAM-FIRST STAGE CENTRIFUGE

FIGURE 26

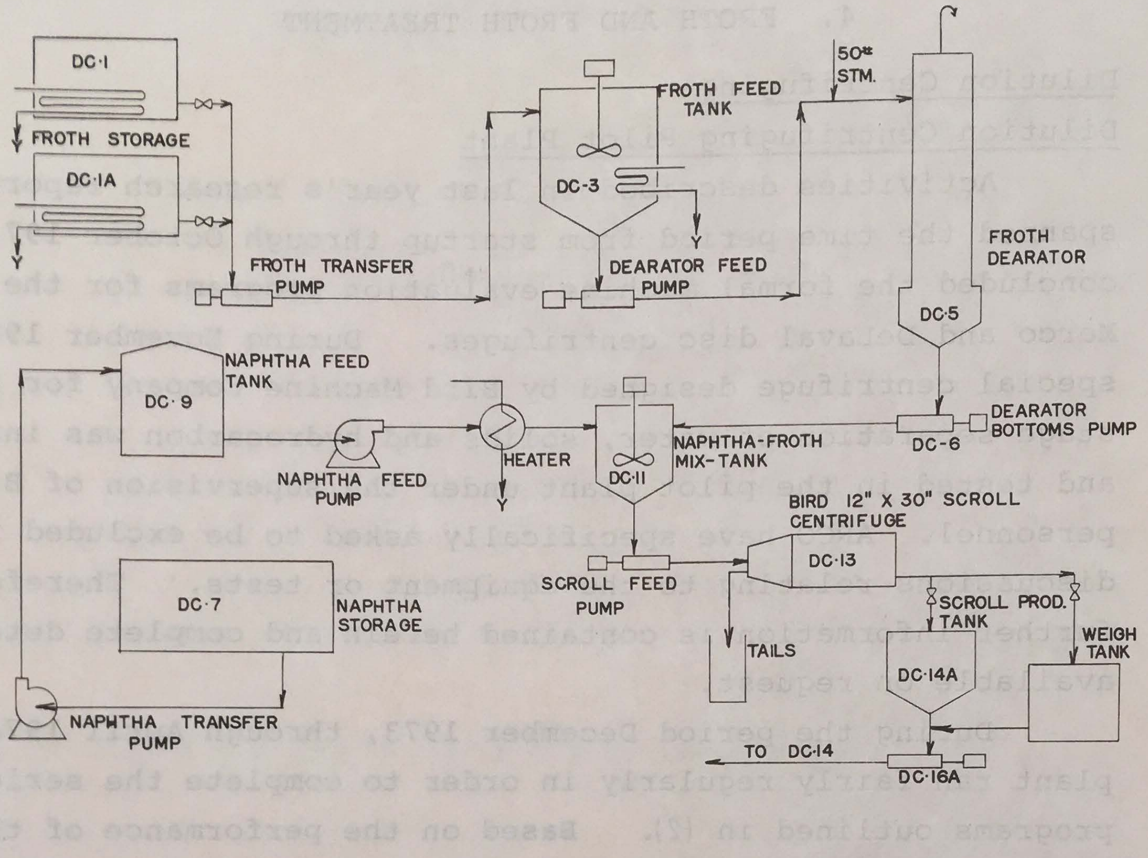
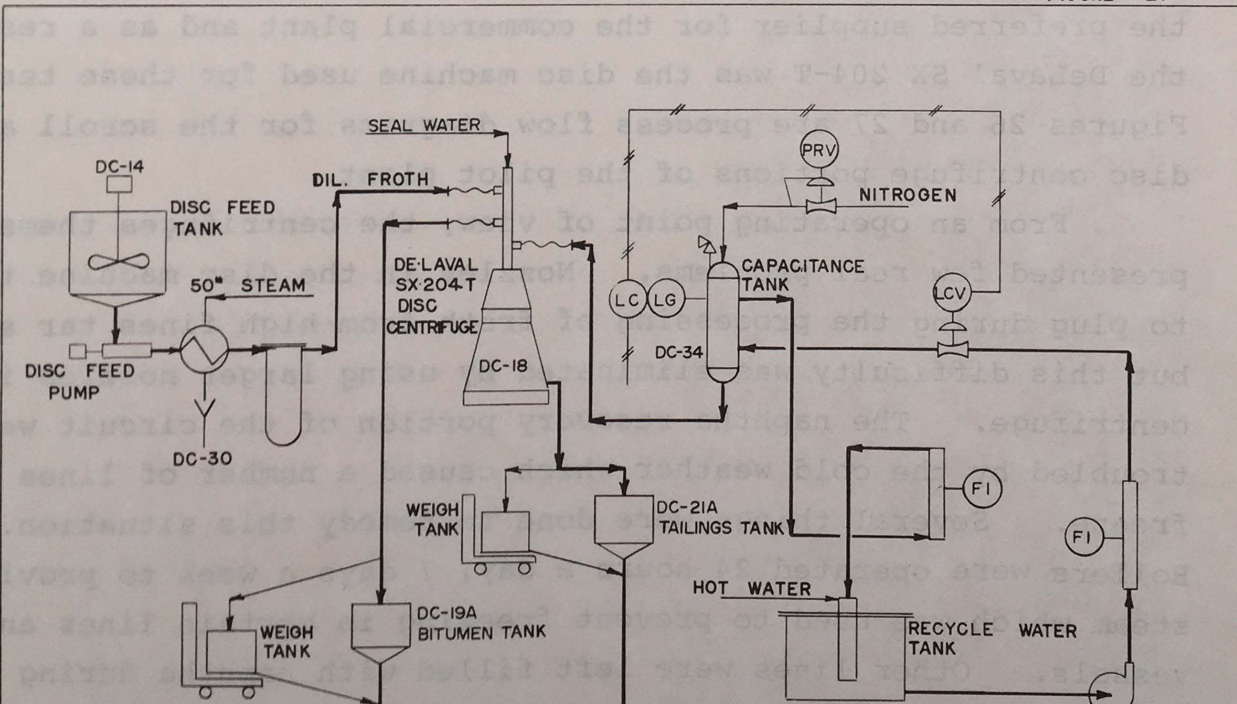


FIGURE 27



FLOW DIAGRAM DE LAVAL SX 204-T

2000 psi pressure to establish a small flow through each coil followed by a controlled burn out with steam and air. The froth storage tanks required cleaning due to sludge accumulation at the bottom of the tanks.

Since April, the pilot plant has operated on an irregular basis primarily to produce tailings for dilution centrifuging loss reduction studies. A large portion of these tailings have been used in testing a gas flotation step which produces a hydrocarbon rich froth. Recently small quantities of this have been batched through the pilot plant.

A number of special studies, not directly related to centrifuge performance but rather to the design and operation of the commercial facilities, were undertaken at the request of the Engineering Department. These included tests on the pulpability of scroll tailings (3), froth deaerator performance at various feed rates for specified feed and bottoms temperatures (4,5,6) and an evaluation of a pipeline viscometer for controlling the naphtha/bitumen in the scroll feed (7,8).

T.E. Kizior
G.R. Lorenz
K.C. Porteous

<u>References</u>

1. Research Department Research Activity 1973, January ,1974, p. 21.
2. "Dilution Centrifuging Pilot Plant Program", Syncrude Interoffice Correspondence, T.E, Kizior and K.C. Porteous to R.R. Goforth, November 23, 1973.
3. "Pulpability of Bird Tailings", Syncrude Interoffice Correspondence, Ref. No. 4439, W.J. Lavender to K.C. Porteous, April 16, 1974.
4. "Froth Heater Deaerator Tests", Syncrude Interoffice Correspondence, Ref. No. 4010, W.J. Lavender to K.C. Porteous, April 5, 1974.
5. "Froth Deaerator Test Result", Syncrude Interoffice Correspondence, Ref. No. 5120, G.R. Lorenz to T.E. Kizior, May 13, 1974.
6. "Supplementary Froth Heater/Deaerator Tests", Syncrude Interoffice Correspondence, Ref. No. 6240, W.J. Lavender to K.C. Porteous, May 24, 1974.
7. "Scroll Centrifuge Feed Viscometry", Syncrude Interoffice Correspondence, Ref. No. 5355, W.J. Lavender to K.C. Porteous, May 8, 1974.
8. "Special D.C. Runs forViscosity and O/W/S Analyses", L.R. Mah to R.E. Hoyle, July 29, 1974.

CENTRIFUGAL AND MOYNO PUMP TESTS

FIGURE 28.

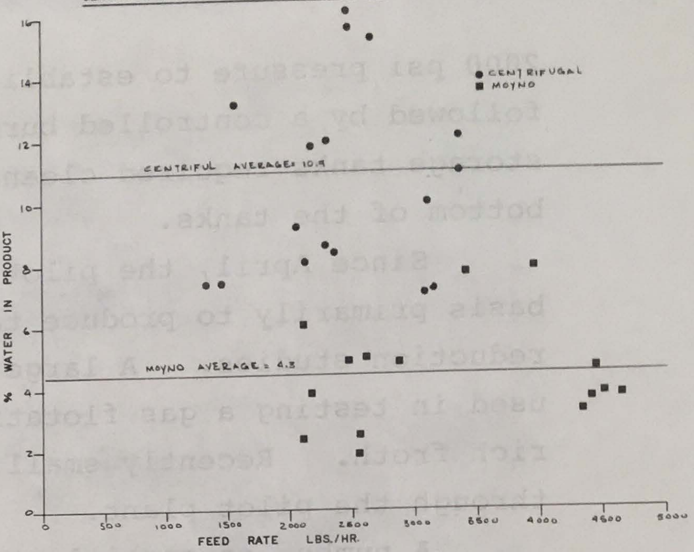
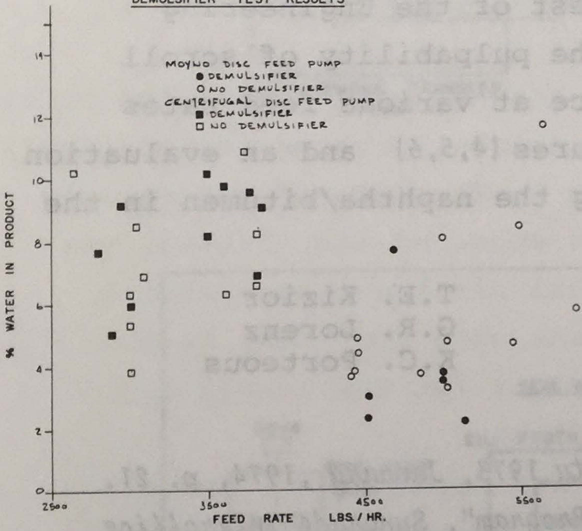


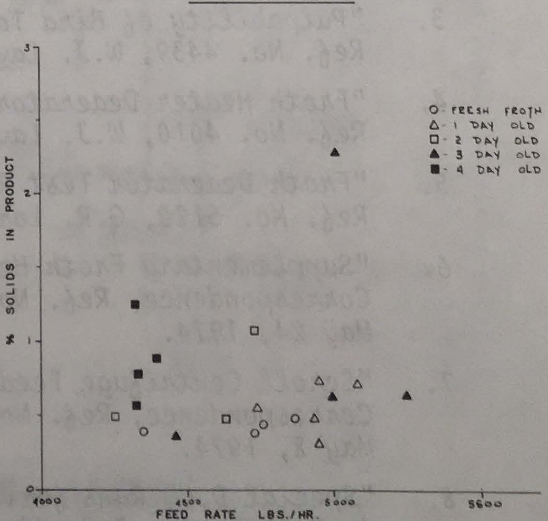
FIGURE 29.

DEMULSIFIER TEST RESULTS



FROTH AGING TESTS

FIGURE 30.



4.1.2 Analyses of Dilution Centrifuging Pilot Plant Performance

Results of special test programs described in (1)have been assessed and will be incorporated in the final dilution centrifuging pilot plant report currently in preparation. The major findings of each study are summarized below:

1. High Temperature Tests

A small number of disc centrifuge tests were conducted at an operating temperature of 200°F and were compared with tests at the normal temperature of 170°F. There did not appear to be any quantifiable improvement in performance at the higher temperature.

2. Demonstration Run

A 100 hour disc centrifuge demonstration run was undertaken with froth produced from high fines tar sand. Although this froth contained considerably more water than that from regular tar sand, the water and solids levels in the diluted bitumen product and the hydrocarbon losses were typical of results with regular froth. Over the range of feed rates examines, there was no discernable effect of feed rate in machine performance.

3. Centrifugal Pump Tests

G.C.O.S. pilot plant experience paralleled our own in respect to achieving good water separations in the disc machine without the use of demulsifying chemicals. However, commercially, they have found that demulsifiers are essential. In both pilot plants Moyno pumps were used to feed the centrifuges, whereas centrifugal pumps are used in the G.C.O.S. commercial operation. Therefore, a series of tests were undertaken in which the disc centrifuge was fed by a centrifugal pump. Unfortunately, the pump was oversized and a significant pressure drop was taken across a valve in the pump discharge line. Figure 28 shows that the centrifugal pump-valve combination resulted in a disc centrifuge product which was wetter than that produced with a Moyno pump. The amount of emulsification resulting from the valve as opposed to the pump is not clear; however, subsequent tests with other centrifugal pumps established some impeller top speed criteria for satisfactory disc performance. These results clearly demonstrate that the manner in which diluted froth is handled can strongly influence the ease of separation.

Table 11

Disc Centrifuge Performance: Gas Sparging Tests
Average Results

Sparging Gas	Product Assay % Water	Product Assay % Solids	% Solids Class'n	% Water Separation	Total Hydro-carbon Loss
None	5.27	0.40	77.1	79.0	0.60
Carbon Dioxide	2.00	0.38	81.2	93.2	1.12
None	3.23	0.40	82.4	87.7	0.82
35% Carbon Dioxide 65% Nitrogen	5.33	0.35	81.5	79.2	0.58
Carbon Dioxide	3.68	0.36	81.2	84.8	0.84

4. Demulsifier Tests

The demulsifier used by G.C.O.S., Alchem D 197, was tested in the pilot plant for both Moyno and cetrifugal disc centrifuge feed pumps. Figure 29 shows the water level in the disc centrifuge product with and without demulsifier. This chemical did not improve disc machine performance with the centrifugal feed pump; however, the water in the final product water may be slightly lower in the case of the Moyno pump.

5. Froth Aging Tests

Froth from a single extraction run was processed in the dilution centrifuging circuit on successive days in an effort to quantify the effect of froth age. Scroll centrifuge performance was independent of age, and the hydrocarbon loss and water content of the proudct from the disc machine were also unaffected. Figure 30 depicts the solids level in the disc product for froths of various ages. There is some indication from these data that the solids separation is more difficult with aged froth.

6. Carbon Dioxide Tests

A combustion gas containing nitrogen and carbon dioxide was proposed as the inert blanketing gas in the commercial plant and there was some concern that the carbon dioxide might make the water and solid separations more difficult, i.e., cause a more stable emulsion. Some laboratory experiments (1) suggested that this gas aided separation and in fact reduced hydrocarbon loss. Work in the pilot plant consisted of sparging carbon dioxide into the scroll product contained in the disc centrifuge feed tank prior to centrifuging in the disc machine. The data, which are shown in Table 11, do not show any consistent effect of carbon dioxide on the process.

7. Diluent Tests

The diluent used for the entire pilot plant program was raw delayed coker naphtha from G.C.O.S. Two other types of naphtha, one a hydrotreated G.C.O.S. naphtha and the other a fluid coker naphtha from a refinery in Billings, Montana, were also evaluated. No significant performance differences were found for these naphthas. Their performance was consistent with that obtained using the raw

delayed coker naphtha which was the diluent used in all other testing.

T.E. Kizior
K.C. Porteous

Reference

1. "Effect of Carbon Dioxide on Diluted Froth", Syncrude Interoffice Correspondence, K.C. Porteous, December 19, 1973.

4.2 Dilution Centrifuging Loss Reduction

4.2.1 Tailings Flotation

Pilot scale flotation of 400 gallon batches of combined disc and scroll centrifuge tailings was first attempted in 1973 (1). The three cell co-current bank of Denver type flotation cells used, typically gave low hydrocarbon recovery unless chemicals or acids were added to the flotation feed.

Analysis of the results of these tests suggested that hydrocarbon recovery, at least as high as with chemical or acid addition, could be obtained by modified flotation conditions (2), namely:

1. Longer residence times
2. Increased agitation
3. Increased aeration

The incentive for these tests was to obtain satisfactory performance without chemical addition. Whereupon, the process would be considerably more economic and a potential source of contamination of the tailings pond eliminated (2).

The first tests at more intense flotation conditions were conducted in August, 1974. Within the accuracy of the analytical methods the recovery was 100%, much better than had been anticipated (3).

Other aspects of the D.C. tailings flotation process which have been investigated are as follows:

1. Maxwell Cell (4)

A Maxwell cell proved to be unsatisfactory for tailings cleanup due to design limitations. The possibility of using a Maxwell cell in this process was investigated because of the potential economic benefits of the much larger cells. The Maxwell

cell is designed for "low intensity" flotation (low aeration and agitation) in contrast to the intense conditions which were found to be most satisfactory for tailings flotation. Within the range of "conventional" cells, the Denver or Wemco type could be suitable.

2. Use of nitrogen as Flotation Gas

Tailings flotation cells in the commercial plant would require an inert flotation gas due to the explosion hazard presented by naphtha vapors. No detrimental effect was found when nitrogen was used as the flotation gas. A mixture of nitrogen/carbon dioxide gases may also be tried as it is the cheapest commercially available form of inert gas.

3. Froth Cleaner (5)

The 12 inch diameter froth cleaner previously used in the secondary recovery circuit of the extraction pilot plant was tested with tailings froth in an effort to reduce its water and solids content. The best performance obtained was about a 10% (absolute) reduction in the froth water content with negligible oil losses or solids rejection. More tests may be conducted if froth water content proves to be excessive for subsequent treatment, otherwise, the cleaner will be eliminated from the flotation circuit.

Further tests are planned to optimize the equipment and conditions for scale-up to a commercial circuit.

The tailings flotation circuit has demonstrated the capability of consistently recovering 98% of hydrocarbons lost in dilution centrifuging tailings. The resulting froth typically contains about 25% hydrocarbon, 50% water, and 25% solids when a froth settler is not used or 30% hydrocarbon, 40% water and 30% solids with a settler. This represents about a 95% rejection of the water content and an 80% rejection of solids from the dilution centrifuging tailings. Further processing of this "dirty" froth is necessary to return the hydrocarbon to the dilution centrifuging product stream (see Section 4.2.2)

L.J. Falkenberg
S.J. Lane

References

1. *Research Department Research Activity, 1973. January, 1974. p.27.*
2. *Research Department Progress Report, August 1, 1974, p.8.*
3. *Research Department Progress Report, September 1, 1974, p.21.*
4. *Research Department Progress Report, October 1, 1974, p.25.*
5. *Research Department Progress Report, November 1, 1974, p.28.*

4.2.2 Treatment of Tailings Flotation Froth

The viability of the flotation process for recovery of hydrocarbons from dilution centrifuging tailings depends on an effective method of removing the water and solids from the recovered hydrocarbon. It had originally been planned to recycle this froth to the front end of the dilution centrifuging operation for mixing with extraction froth. However, there are considerations which warrant processing this froth separately. Recirculation of tailings froth might create a problem of solids building up in the dilution centrifuging circuit and result in poorer separations. This build up seems probable because of the predilection of solids, such as heavy minerals, to float.

A process of diluting the froth with naphtha and centrifuging, similar to dilution centrifuging, was found to give 95% recovery of the hydrocarbons on a laboratory scale (*1*). To test it on the pilot scale, several barrels of froth were produced using the flotation unit. In the first pilot plant tests the froth was allowed to cool and settle. Some water was drained off. The froth was diluted with naphtha to give about a 1:1 naphtha/bitumen ratio and processed at various feed rates through both disc and scroll centrifuges (*2*). Pilot scale hydrocarbon recovery was 86-92%, but problems of sludge accumulation in the disc machine caused operating difficulties. When the froth was kept at the temperature of the flotation cells and processed soon after flotation using large nozzles in the disc centrifuge, no difficulties arose because of sludge accumulation, but water "breakout" from the scroll product in the disc feed tank resulted in a non-homogeneous feed to the disc machine. Two approaches to this problem are now being

investigated. Either a better quality froth will be produced from the flotation step, if possible, or water and solids will be removed by gravity settling prior to disc centrifugation.

The froth treatment process has achieved a 90% recovery of the dilution centrifuging hydrocarbon losses under conditions feasible for commercial operation. This would mean the overall hydrocarbon recovery of dilution centrifuging commercially would be raised from 98.0% to 99.8%. The best product quality of the additional hydrocarbon recovered was:

Hydrocarbon (N/B = 1/1)	Water	Solids
96.14	3.22	.64

This product would have a negligible impact on the quality of dry bitumen fed to the fluid coker.

R.W. Ashworth
S.J. Lane

References

1. Research Department Progress Report, November 1, 1974, p.33.
2. Research Department Progress Report, December 1, 1974, p.33.

4.2.3 Naphtha Flashing

Laboratory test work was carried out to determine the feasibility of using a vacuum flash technique for recovering naphtha from dilution centrifuging tailings. Bechtel (1) had shown that such a scheme was theoretically possible and economically feasible. Three pieces of apparatus were constructed, the first measured vapor pressures (1,2,3), the second apparatus (4) was used for batch vacuum distillations, and the third apparatus (5) provided the capability for a continuous vacuum flash experiment.

From the start of the test program, it was evident that the combined scroll and disc tailings from the dilution centrifuging pilot plant did not behave according to the theoretical flash calculations. The hydrocarbon vapor pressure and recovery were much lower than anticipated. Under the same flashing conditions, the scroll tailings yielded significantly different results than

disc tailings. Scroll tailings gave 30-50% (6) naphtha recoveries compared to the theoretically predicted recovery of 55% of the feed naphtha at the conditions proposed for a commercial scheme (i.e., 5 psi and 155°F). However, the disc tailings, which should have given similar recoveries, consistently yielded values of 5% or less. At first, it was thought that naphtha assays on the disc tailings were possibly erroneous and that disc tailings actually contained little or no naphtha. However, measurements of this naphtha content were made using a gas chromatograph and these essentially confirmed the naphtha assay by the normal analytical technique. The reason for this unexpected vapor-liquid equilibrium behaviour is unknown but it makes the flashing scheme much less economically attractive.

Although some additional work is planned on the flashing, the current emphasis is on recovery of tailings bitumen via inert gas flotation (7). In the course of this process, the flotation gas strips naphtha from the tailings and it appears that naphtha recoveries comparable to the flashing scheme are achievable by condensing naphtha from the gas stream.

R.W. Ashworth
S.J. Lane

References

1. *Research Department Progress Report, March 1, 1974, p. 16.*
2. *Research Department Progress Report, April 1, 1974, p. 7.*
3. *Research Department Progress Report, May 1, 1974, p. 7.*
4. *Research Department Progress Report, June 1, 1974, p. 30.*
5. *Research Department Progress Report, July 1, 1974, p. 22.*
6. *Research Department Progress Report, October 1, 1974, p. 19.*
7. *Research Department Progress Report, November 1, 1974, p. 33.*

5. BITUMEN CHEMISTRY

5. BITUMEN CHEMISTRY

5.1 Evaluation of Core Hole Bitumens

Bitumens extracted from a core from the mining area in Lease 17 were evaluated with the following objectives:

1. To determine the variability of bitumen from one location and variable depth.
2. To correlate bitumen characteristics to the nature of the mineral matrix.
3. To determine Conradson Carbon as a function of depth.
4. To determine distillate yield as a function of depth.

The core selected (core number 37-12-1) was of such a nature that the second objective could not be pursued since the tar sand was uniformly rich with virtually no barren layers.

The core hole was sampled along its depth in 12 intervals each 5 ft long. After extraction with cold toluene and settling of the extract in a two stage continuous settler the toluene was distilled off and the remaining bitumen analyzed for:

a) residual toluene
b) ash
c) Conradson Carbon Residue
d) Wt % distillate <975°F
e) S.A.R.A. (Saturates, Aromatics, Resins, Asphaltenes)
f) Elemental analysis: C, H, S
g) Specific Gravity (in doubt because of the residual toluene)
h) Absorbance at 600 nm
i) Thermogravimetric analysis at 10°C/min.

The detailed findings are available (1).

The tar sand was rich; the weighted average bitumen content for the hole was 14.11 wt %. In general the distillate content decreases by as much as 6 wt % with increasing depth. Two explanations could be given for this phenomenom. First, heavy oil components have settled towards deeper levels and secondly, lighter components are lost from the top of the deposit and are replenished from the bottom, the observed distribution is then comparable to the tail of a gas chromatographic peak. It is however, necessary for this second explanation to postulate some sort of carrier, gas or liquid.

Conradson Carbon Residue values range from 11.7 to 13.3 with an average of 12.4. Considering the uncertainty in one determination of CCR, these values are well within the design limits of the fluid coker.

No significant trends were observed in the elemental analysis. Of analytical interest is the absorbance of the bitumens at 600 nm. These varied between 0.336 and 0.425 A lg^{-1} which indicates that analytical techniques based on an assumed constant value of the absorbance should be used with caution (2). The observed absorbances correlate significatnly with the asphaltene content of the bitumens, the latter varying between 14.1 and 17.9 wt %.

When plotting many of the data as a function of depth it becomes clear that, indeed, the McMurray formation does consist of three members, a top, middle, and bottom one, each of them showing distinct trends and values for the parameters investigated. As such this observation fulfills the second objective of the study in that correlation between the geological description and the composition of bitumen seems to exist.

R. Schutte

References

1. *A tabulation of all the data is available in the Research Department Library.*

2. *Patel, M.S. "Rapid and Convenient Laboratory Method for Extraction and Subsequent Spectrophotometric Determination of Bitumen Content of Bituminous Sands". Anal. Chem., 46 (6) 794 - 795, (1974).*

5.2 Oxidation of Bitumen Components

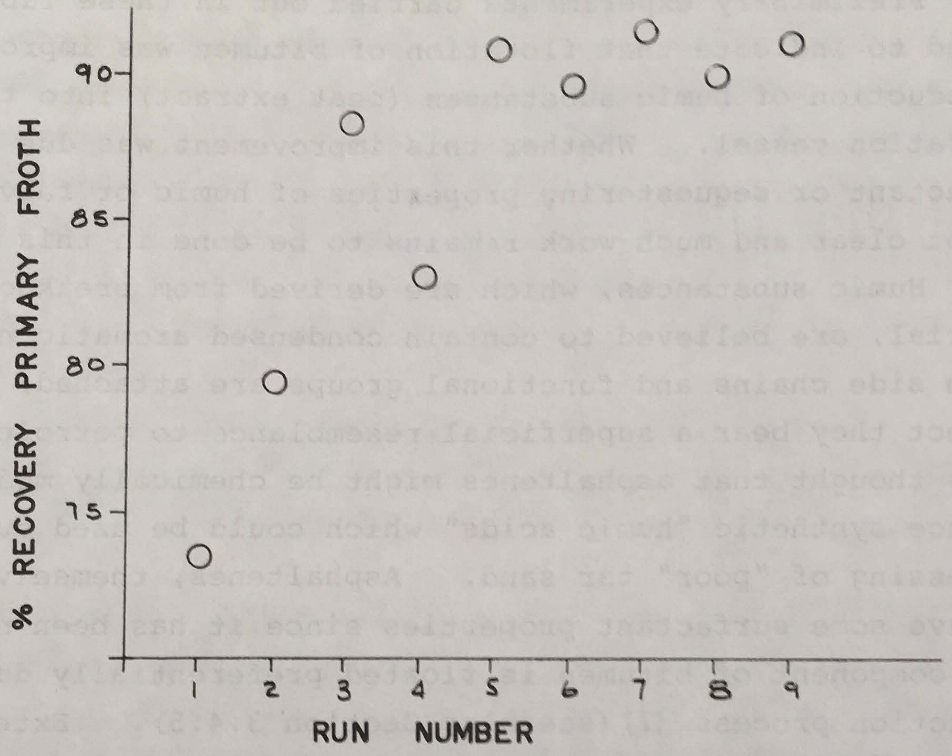
Preliminary experiments carried out in these laboratories seemed to indicate that flotation of bitumen was improved by the introduction of humic substances (peat extract) into the primary separation vessel. Whether this improvement was due to the surfactant or sequestering properties of humic or fulvic acids is not clear and much work remains to be done in this area.

Humic substances, which are derived from breakdown of plant material, are believed to contain condensed aromatic networks to which side chains and functional groups are attached; in this respect they bear a superficial resemblance to petroleum asphaltenes. It is thought that asphaltenes might be chemically modified to produce synthetic "humic acids" which could be used during the processing of "poor" tar sand. Asphaltenes, themselves, appear to have some surfactant properties since it has been noted that this component of bitumen is floated preferentially during the extraction process (1) (see also Section 3.4.5). Extensive studies on asphaltene chemistry have already been carried out by Speight at Alberta Research (2), and it is known that surfactants may be generated by the introduction of sulfonate groups. Experiments have also been carried out in these laboratories in which samples of sulfonated bitumen were added to tar sand in the stainless steel pot (3).

The introduction of sulfonate groups into a material already high in sulfur content is, however, a retrograde step since the additional sulfur must be removed at a later stage. Since, in general, sulfur is easier to oxidize than carbon, a possible alternative may be to convert (by mild oxidation) the 6 to 8% of sulfur already present in asphaltenes into useful functional groups. Oxidation of some sulfur compounds obviously occurs during the extraction process since sulfoxides and sulfones have been detected in aqueous process streams (4).

RESULTS OF 500 g BATCH EXTRACTION EXPERIMENTS

FIGURE 31.



Run Number	Additives	Oil Recovery as a Percentage of Total Oil Recovered from S.S. Pot: Primary Froth	Secondary Froth	Total Froth
1	0.8g Asphaltenes I *	73.58	13.67	87.25
2	Blank	79.58	7.64	87.22
3	3ml 1N NaOH	88.40	3.0	91.40
4	3ml 1N NaOH 0.1g Asphaltenes	83.10	8.02	91.12
5	3ml 1N NaOH 0.1g Asphaltenes I	90.91	2.3	93.21
6	3ml 1N NaOH 0.1g Asphaltenes II **	89.62	3.4	93.02
7	3ml 1N NaOH 0.8g Asphaltenes	91.41	2.2	93.61
8	3ml 1N NaOH 0.8g Asphaltenes I	89.91	3.5	93.41
9	3ml 1N NaOH 0.8g Asphaltenes II	91.00	2.1	93.10

*Asphaltenes I-oxidized 6 hr.

**Asphaltenes II-oxidized 24 hr.

In summary the findings are as follows:

1. <u>Oxidation Studies</u>

It is probable that the majority of the sulfur contained in asphaltenes is present in an aromatic (thiophenic) environment. Unfortunately, conditions normally employed for the oxidation of thiophenic compounds proved to be too severe for this system (5), and lead to extensively polymerized products.

Oxidations carried out under milder conditions were more successful; the action of air on a hot stirred suspension of pentane asphaltenes gave rise to two main fractions (6).

The major fraction, designated as oxidized asphaltenes, was insoluble in sodium hydroxide solution and closely resembled the original asphaltenes. The IR spectrum of the sample, however, confirmed the oxidized nature of this material.

The sodium hydroxide soluble fraction yielded, on acidification, a brown amorphous substance similar in appearance to precipitated humic-acid. For this reason the fraction was termed "synthetic humic acid".

2. <u>Experiments using the Laboratory Batch Extraction Unit</u>

The effectiveness of generated chemicals as flotation aids for the extraction process was tested using the 500 g batch extraction unit (7). An "average" tar sand was used for the studies and effectiveness was measured in terms of primary recovery. Asphaltenes, and two samples of oxidized asphaltenes (differing in the extent of oxidation) were added as dry powders at the slurry stage.

It was demonstrated that the added chemicals were recovered in the primary froth (7). Since the additives were analyzed as "oil", all recoveries were expressed as a percentage of the total recovery from the 500 g unit. It may be seen from the accompanying Figure 31 that each of the additives produces a marginal increase in primary oil recovery. This increased recovery is, however, only achieved in the presence of sodium hydroxide.

J.E. Filby

References

1. *Research Department Progress, February 1, 1973, p. 24.*
2. *S.E. Moschopedis and J.G. Speight, "Oxidation of Bitumen in Relation to its Recovery from Tar Sand Formations", Fuel, 53, 21 (1974).*
3. *Research Department Research Report, 1968, p. 77, Syncrude Canada Ltd., Report S-2M-68.*
4. *Research Department Research Report, 1968, p. 70, Syncrude Canada Ltd., Report S-2M-68.*
5. *Research Department Progress Report, May 1, 1974, p. 11.*
6. *Research Department Progress Report, June 1, 1974, p. 1.*
7. *Research Department Progress Report, July 1, 1974, p. 1.*

5.3 Thermal Cracking of Bitumen

Research into the basic properties of Athabasca bitumen continued during 1974. The three-phase flow through thermal reactor system as described in reference(1) was completed and run successfully. This system is capable of thermally cracking bitumen, containing up to 8 wt % solids, at a maximum capacity of 8 liters per hour. Considerable conversion of non-distillable material to distillate and gas was achieved without plugging or significant coke laydown.

The material balances around the unit achieved closures of about 95%. The main reasons for these poor balances have been identified. They are the malfunctioning of the wet test meters and an excessive holdup in the high pressure separator downstream from the reactor. These conditions are being corrected.

A report describing the details of the operation and analytical procedures, and giving feedstock and product inspections of the run, is in the final stages of preparation.

Future Activities

Future work includes the addition of stripping gases (nitrogen and steam) to the reactor, and the conversion of the unit to a hydrocracker by virtually replacing the stripping gases by hydrogen. Some modifications to the building are underway to accommodate a hydrogen containing reactor system.

R. Schutte

Reference

1. *Research Department Research Activity, 1973. January, 1974, p. 33.*

5.4 Polycyclic Aromatic Hydrocarbons

An investigation into the possible long-term hazards associated with the handling of Athabasca bitumen was conducted earlier (1). The concentration of certain carcinogens (polycyclic aromatic hydrocarbons) was found to be low and, by analogy to materials of similar composition, it was concluded that the risk of skin cancer resulting from handling of this material was slight.

Because the results of this study were felt to be of sufficient importance to warrant outside publication, further work was recently carried out to (i) expand the available data and (ii) verify the results of the study for several bitumen types.

The results of the recent work fully support those of the earlier study, even though the analytical methods used were different. The two bitumens examined were similar and neither differed appreciably from the sample examined in the previous study.

J.E. Filby

Reference

1. *Filby, J.E., Endres, J.D., and Schutte, R. "Polycyclic aromatic hydrocarbons in Athabasca bitumen". April, 1974.*

5.5 Tar Sand Pyrolysis

Direct coking has long been proposed as a method for the production of oil from the Athabasca tar sands, and extensive work has already been carried out concerning the direct fluid coking of this material (1) (see also Section 3.4.5). More recently, in view of the undesirably high water requirements of conventional extraction processes, the topic has been the subject of renewed interest (2,3).

A discussion on the subject of tar sand coking may be found in the recent Seyer report (4) (see also Section 3.4.3) From an energy-balance and economic standpoint, tar sand coking may be an attractive alternative to conventional extraction and upgrading methods.

TABLE 12

Coke Yields from Tar Sand Pyrolysis (TGA Studies)

	Hydrogen Atmosphere		Nitrogen Atmosphere	
	1	2	1	2
Coke yield from tar sand 1	8.79%	8.99%	7.35%	8.76%
Coke yield from tar sand 2	10.63%	10.91%	9.36%	8.53%
Coke yield from tar sand 3	8.10%	8.34%	8.45%	9.03%

Footnotes:

1. Temperature during a) pyrolysis, 800°C (rate of heating, 160°C min^{-1}).
   b) ashing, 700°C.
2. Tar Sand 1: 12.68% oil, 4.98% water, 82.34% solids
   Tar Sand 2: 8.33% oil, 6.37% water, 85.31% solids (PSD (-325) 19.94%)
   Tar Sand 3: 13.25% oil, 0.28% water, 86.48% solids (PSD (-325) 7.80%)
3. Effect of heating rate on tar sand pyrolysis

Tar Sand 3 Nitrogen atmosphere 800°C	Rate of Heating (°C min)	Sample Weight (mg)	% Weight Loss
	40	11.72	12.63
	40	12.54	12.68
	80	12.00	13.08
	80	13.88	12.68
	80	13.78	12.77
	160	13.52	12.65
	160	13.40	12.54
	160	11.70	12.82

TABLE 13

Pyrolysis of Tar Sand in Tube Furnace

Run Number	5	6	7	8	9*
Charge Weight (g)	1.7220	1.5588	1.9468	1.9966	2.1142
Purge Gas	N_2	N_2	N_2	N_2	N_2
Purge Flow (ml/min @ STP)	96.2	97.8	98.0	97.5	101.4
Wt% Total Product	12.46	11.82	11.85	11.70	12.11
Wt% Residue	(not available)	87.46	87.37	87.36	87.11
Wt% Liquid Product	82.26	79.76	81.16	81.00	83.45
Wt% Ash	85.50	85.99	85.92	86.01	85.85
Wt% Coke	10.83	11.14	10.97	10.19	8.78
Wt% Loss	0.31	0.72	0.79	0.94	0.74

Run Number	10	11	12	13	14	15
Charge Weight (g)	1.8247	1.8210	1.7209	1.9508	2.2349	1.9752
Purge Gas	H_2	H_2	N_2	N_2	N_2	N_2
Purge Flow (ml/min @ STP)	98.8	94.5	199.7	198.8	300.4	304.0
Wt% Total Product	11.56	11.79	12.20	11.88	11.92	12.40
Wt% Residue	87.23	87.19	87.38	87.53	86.00	87.09
Wt% Liquid Product	84.75	86.87	79.51	83.43	81.64	87.99
Wt% Ash	85.88	86.04	86.09	86.26	84.52	85.71
Wt% Coke	10.22	11.19	9.74	9.62	11.14	10.32
Wt% Loss	1.21	1.02	0.42	0.59	2.08	0.51

Run Number	16	17	18
Purge Gas	N_2	N_2	H_2
Bromine Number	50	40	58

Footnotes:

1. Weight percentage liquid product and coke are expressed on the basis of the oil content (as determined by O/W/S). Other weight percentages are expressed on the basis of sample weight (tar sand).
2. Final Temperature: 800°C, Time at this temperature; 18 minutes
   Heat up time, 12 minutes
   Except * final temperature 1000°C, heat up time 22 minutes

A preliminary investigation into the pyrolysis of tar sand was, thus, undertaken. It was felt that sufficient information would be obtained from laboratory scale experiments to at least indicate whether or not further work could be justified.

1. Thermogravimetry Experiments

Initial experiments were carried out using a thermobalance; temperature, heating rate and atmospheric conditions may readily be controlled using this apparatus but the technique is limited to very small samples (15-20 mg). Difficulties were experienced at first in obtaining representative samples of tar sand of this size, however, a reliable technique for the small scale homogenization of tar sand was developed (3).

A summary of the results of pyrolysis studies on three tar sands is shown in Table 12. Since it was of interest to investigate the effect of hydrogen on coke yield, experiments were carried out in nitrogen and hydrogen atmospheres. Coke formed during the pyrolysis was weighed and then burned off by continuing the TGA experiment in air. Differences between the residue weight in hydrogen/nitrogen and the ash yield (in air) were assumed equal to the coke yield.

Samples of isolated bitumen and tar sand solids are examined separately. Coke yields from bitumen alone are consistent with the results of the tar sands experiments indicating a negligible kinetic effect due to the solids. The small weight losses observed for the tar sand solids indicated the presence of only small quantities of chemically bound water.

Initial indications were that coke yields decreased as the final temperature was increased, although differences between coke yields at 800°C and 950°C were found to be small. The majority of the experiments, then, were carried out at 800°C to maximize furnace life.

At this final temperature, coke yields appear to be quite low. In view of the small weight differences involved, the coke and product weight yields from experiments carried out in hydrogen and in nitrogen, are not significantly different. Residue yields were not markedly affected by the rate of heat-up.

These results are encouraging and indicate that more work in this area is warranted. The results, however, must be interpreted with some caution. The experiments provide, for example, no information as to the relative yields of gaseous and liquid products. These could conceivably be unfavourable at the temperature of these experiments.

2. Pyrolysis Tube Experiments

Tar sand samples (1-2 g) were heated electrically in a tube furnace (5). Volatile products, swept out of the tube by a constant flow of purge gas (hydrogen or nitrogen), were collected in traps cooled to -78°C. In this way an estimate of liquid product yield could be made.

Coke yields were determined using the same technique employed for the TGA experiments, by burning off the coke from the residue and reweighing.

The results of several experiments carried out using this apparatus are shown in Table 13. Liquid product yields were calculated, in each case, as a percentage of the oil present in the sample.

Liquid product yields are encouraging, greater than 80% in all cases. This is high but in line with Gishler's (1) results. The coke yields are, however, a little higher than those determined for the TGA experiments.

The results of the TGA work were confirmed in that hydrogen as carrier gas did not produce an effect on the coke yield

Bromine numbers were determined in liquid products for experiments conducted in both hydrogen and nitrogen (Table 13). This test provides a rough measure of the degree of unsaturation of the sample. The results show that hydrogenation of the product does not occur where hydrogen is used as carrier gas.

J.E. Filby
R. Schutte

References

1. Peterson, W.S. and Gishler, P.E., "Oil from Alberta Bituminous Sands", Petroleum Engineer, 23 (4), 66-74, (1951)

2. "Current Oil Sands Production Methods Gulp and Pollute too Much Water", Canadian Petroleum, 15 (9) 37-38, (1974).

3. Research Department Progress Report, November 1, 1974, p.7.

4. F.A. Seyer, "Technology of Hot Water Processing and Research Needs", September, 1974.

5. Research Department Progress Report, December 1, 1974, p. 1.

6. ANALYTICAL DEVELOPMENT

6. ANALYTICAL DEVELOPMENT

The function of the analytical development group is to develop new or improved techniques and methods as required for the support of the various research programs. Attention is also being given to methods and equipment required for the commercial and environmental field laboratories.

6.1 Analytical Instrumentation

The following is a brief review of this year's activities.

1. Oil/Water/Solids Replacement

The analyses of samples for oil/water/solids content is a continuing major consumer of manpower, space and supplies.

Samples were submitted to Princeton Gamma-Tech for X-ray fluorescence analysis of sulfur as a measure of the oil content. The returned data showed the expected problem, i.e., that the sample examined by the XRF unit was so thin that good data could not be obtained. In addition it showed an extremely strong reciprocal relationship of calcium and oil contents which had not been previously noted (1).

A new infrared reflection type analyzer manufactured by Anacon was investigated. While it showed considerable promise (2) it was obvious that material other than oil and water was affecting the meter readings obtained. Further study is being carried out using the laboratory IR Spectrophotometer to determine the IR absorption characteristics of the various materials encountered, with the hope that a better wavelength selection can be made to further improve performance.

Inquiries have been made about the Broker pulsed NMR equipment for oil and water measurement. There appear to be basic problems preventing application of this equipment, but the topic remains under consideration.

2. Particle Size Analysis

A sedigraph 5000 particle size analyzer (3,4,5,6) has been purchased. This equipment has the following advantages relative to the sedimentation balance method previously employed:

a) Direct plotting of particle size vs. weight percent.

b) Rapid measurement (5 to 10 minutes per sample).

c) Direct measurement of the property of interest, i.e., sedimentation rate in water.

d) High repeatability and accuracy.

e) Reduced operator influence - technique variations from operator to operator have little effect.

The Sedigraph operation is not without difficulties. The advantage relative to previous methods is that most of the operating problems are immediately obvious from viewing the resultant chart, so the problem can be corrected immediately and the sample rerun.

3. <u>X-ray Fluorescence Analysis</u>

Mention has been made of the testing of an XRF method for oil/water/solids analysis.

While XRF is a versatile and widely usable tool for analysis, it is recognized as requiring a rather high capital, space and support cost for a high grade instrument. In the latter part of 1973 the pressing need for more rapid, approximate analyses in the mineral upgrading program resulted in purchase of a Pitchford Industries Porta-Spec. This unit does not have the flexibility or sensitivity we would want in a research unit, but it has provided rapid analysis for screening purposes in the mineral upgrading program (7). Material balance closures in the ± 5% area indicate that the results are good relative values at least. It has also given us hands-on experience in XRF.

4. <u>Density Determinations</u>

Measurement of density, if accurately done, is a means of obtaining bitumen/naphtha ratios on dilution centrifuging streams and other information. Unfortunately, the routine methods previously used were technique sensitive. A recently introduced unit, the Anton-Paar DMA-10 was obtained for evaluation and subsequently purchased (8). A study carried out in the Research laboratory showed that on the first operation, using a written set of instructions, ten technicians were able to obtain densities within 0.001 g/cc on each of four pure materials (9). With the technique sensitivity thus eliminated, work is proceeding on studying the application of density measurements to a number of analytical problems.

A recent published evaluation was in agreement with present findings (10).

5. Environmental Laboratory Support

Support provided for the environmental laboratory involved -

a) Specifications, selection and acceptance checking of the trailer, furniture and initial equipment. (See Section 7.1).

b) Debugging, installation and start-up of the total organic carbon analyzer. The unit was delivered to the Edmonton laboratory and started up by the Beckman service representative. During its use in the water treatment pilot plant work a number of instrumental problems were encountered, which were eliminated. In late fall the unit was sent to the environmental laboratory and assistance provided for the start up at that location by the analytical group.

G.R. Thompson
R.E. Hoyle

6. High Pressure Liquid Chromatography

This technique has the capability of performing separations over the entire boiling range of bitumen and in this respect, complements the gas chromatographs which are restricted to relatively volatile material.

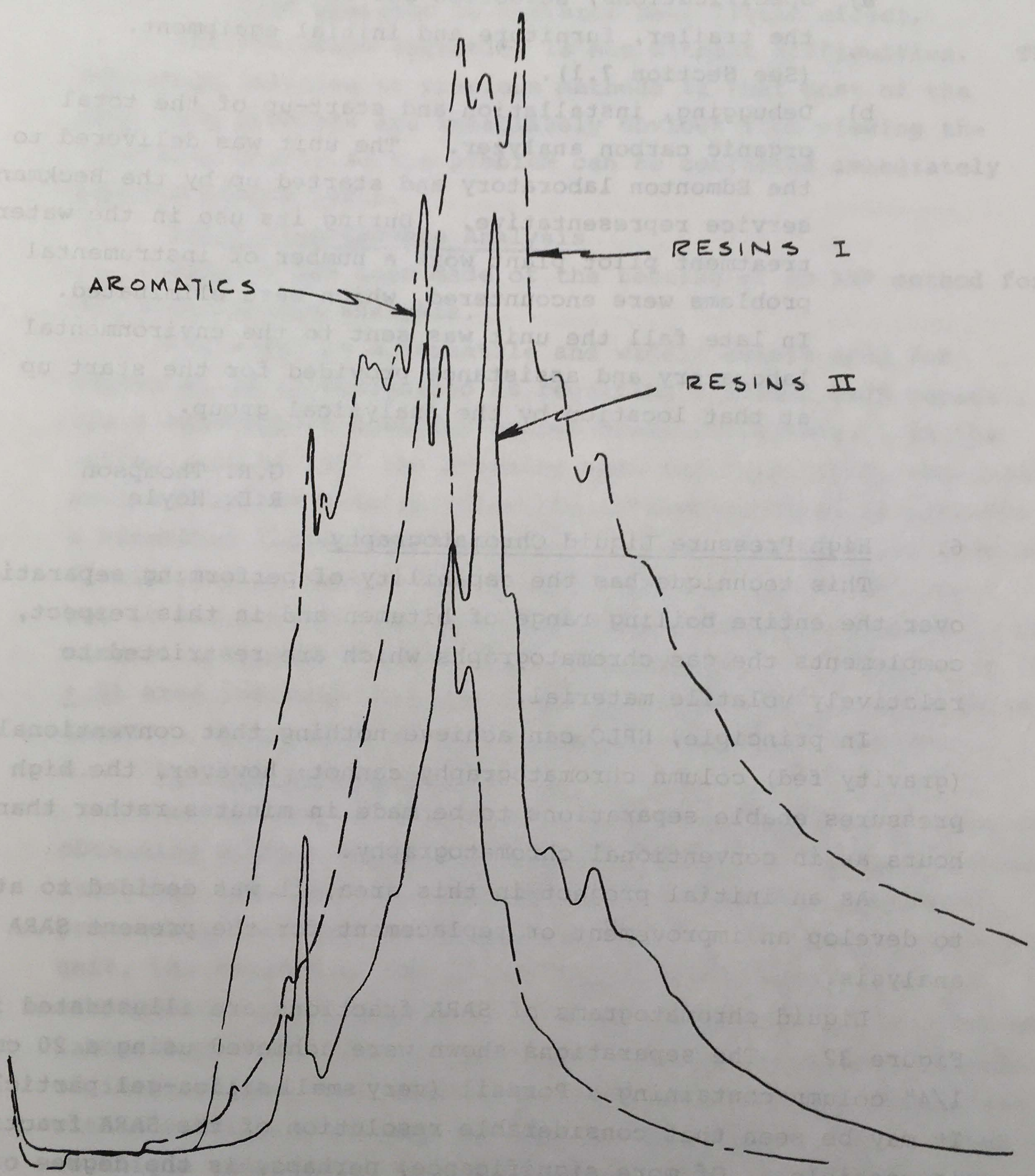
In principle, HPLC can achieve nothing that conventional (gravity fed) column chromatography cannot; however, the high pressures enable separations to be made in minutes rather than hours as in conventional chromatography.

As an initial project in this area, it was decided to attempt to develop an improvement or replacement for the present SARA analysis.

Liquid chromatograms of SARA fractions are illustrated in Figure 32. The separations shown were achieved using a 20 cm x 1/4" column containing μ Porasil (very small silica-gel particles). It may be seen that considerable resolution of the SARA fractions is possible. Of more significance, perhaps, is the degree of overlap between the various fractions, which illustrates the poor resolution of the SARA technique.

ANALYSIS OF SELECTED SARA FRACTIONS

FIGURE 32.



The SARA technique is limited to samples of low volatily and has been used almost exclusively for hydrocarbon-type analyses of bitumen samples. HPLC does not suffer from this disadvantage and could be used (perhaps in conjunction with FIA analysis) for characterization of products from experimental units such as the flow through thermal reactor. Analysis for olefins, for example, would yield information as to the severity of cracking conditions.

J.E. Filby
D. Muir

References

1. *Research Department Progress Report, December 1, 1974, p. 26.*
2. *Research Department Progress Report, September 1, 1974, pp. 13-17.*
3. *Research Department Progress Report, June 1, 1974, p. 22.*
4. *Research Department Progress Report, August 1, 1974, p. 6.*
5. *Research Department Progress Report, September 1, 1974, p. 10.*
6. *Research Department Progress Report, October 1, 1974, p. 14.*
7. *Research Department Progress Report, March 1, 1974, p. 26.*
8. *Research Department Progress Report, May 1, 1974, p. 23.*
9. *Research Department Progress Report, June 1, 1974, p. 18.*
10. *Persinger, H.E., Lowen, J.E., and Tamplin, W.E. "Evaluation of a densimeter", American Laboratory 6 (9) 69-74 (1974).*

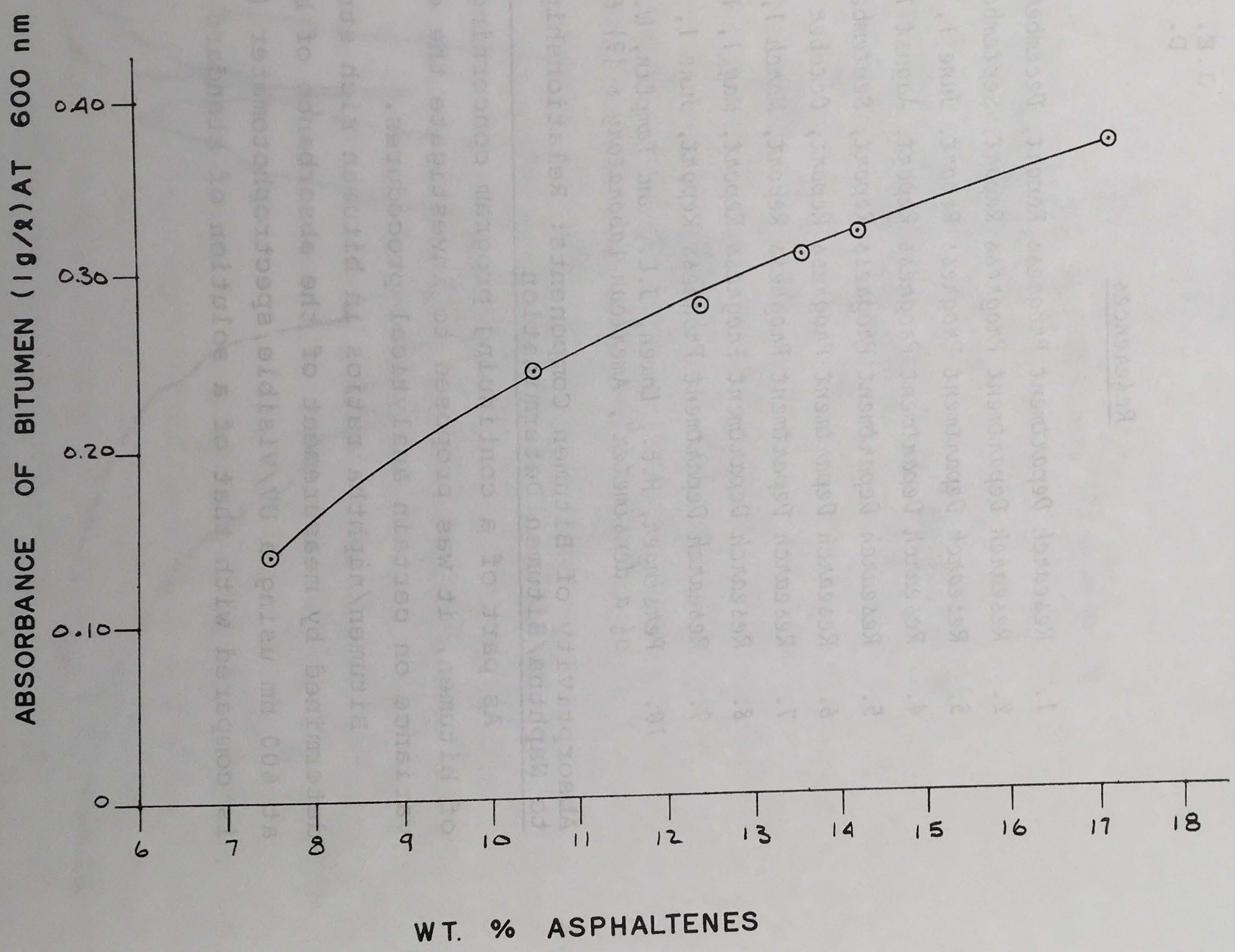
6.2 Absorptivity of Bitumen Components: Relationship to Naphtha/Bitumen Determination

As part of a continuing program concerning the non-homogenity of bitumen, it was proposed to investigate the effects of bitumen variance on certain analytical procedures.

Bitumen/naphtha ratios in bitumen rich streams are presently determined by measurement of the absorbance of bitumen solutions at 600 nm using a UV/Visible spectrophotometer (*1*). This absorbance is compared with that of a solution of standard bitumen.

FIGURE 33.

ABSORPTIVITY OF BITUMEN



Inspection of Table 14 leads to the conclusion that,

TABLE 14

	Absorbance @ 600 nm (1 g/ℓ)	Typical Composition (%)
Saturates	0.000	22
Aromatics	0.000	22
Resins I	0.101	35
Resins II	1.576	4
Asphaltenes	2.030	17

at this wavelength, the absorbance is due almost entirely to the presence of asphaltenes. Since the asphaltene content of bitumen commonly varies between 13 and 17%, this variance will, clearly, have an effect on the absorbance of solutions of bitumen. This fact is illustrated in Figure 33, which shows a linear relationship between absorbance and asphaltene content in the range 10-17% asphaltenes (2).

The "worst case" of a standard of high asphaltene content, together with a sample of low asphaltene content could conceivably result in a naphtha bitumen ratio incorrect by ± 100% (3).

However, providing the limitations of the method are realized, and if bitumen standards are chosen with care, large errors are unlikely to arise during the analysis of diluted bitumen product for the dilution centrifuge.

For samples containing unusually low (or high) concentrations of asphaltenes, such as oil from tailings streams, present spectroscopic methods of analysis are clearly inapplicable; since, for these samples, suitable standards cannot be selected.

A proposed gas chromatographic method for the determination of naphtha has recently been successfully adopted for the analysis of naphtha in DC tailings streams (4).

J.E. Filby
R. Schutte

References

1. *S.A.M. 001/2*
2. *Research Department Progress Report, February 1, 1974, p. 18.*
3. *Research Department Progress Report, February 1, 1974, p. 19.*
4. *"Determination of Naphtha Content of D.C. Tailings", Syncrude Interoffice Correspondence, D. Maxwell to K.C. Porteous, November 4, 1974.*

.3 Refractive Index of Saturates and Aromatics

The SARA procedure separates the petrolene fraction into saturates and aromatics by means of solid-liquid chromatography over alumina and silica gel. This procedure is time consuming. Binary mixtures can, under favourable conditions, be analyzed by measurement of the refractive index *(1)*. The favourable conditions are: a reasonably wide spread between the refractive indices of the components and the absence of volume contraction or expansion on mixing of the components. Volume changes on mixing of saturates and aromatics are not expected so that the measurement of the indices of the components remained to be investigated. An additional result of these measurements would be that some measure of the variability of saturates and aromatics would be obtained.

It was found that the refractive index of the saturates fraction is constant within narrow limits: $n_D^{20} = 1.4850° \pm 0.0004^6$ (95% confidence limits of average 26 samples), while that for the aromatics varies to a greater degree: $n_D^{20} = 1.5559^1 \pm 0.0016$.

On the basis of these results, it is judged possible to determine saturates and aromatics (as defined by the SARA procedure) in the petrolene fraction within about 5% absolute on an original bitumen basis. The losses incurred in the SARA procedure for saturates and aromatics are eliminated by the refractive index method.

The larger limits for aromatics indicate that that fraction is not as well defined as the saturates fraction, This is confirmed by recent liquid chromatography results.

R. Schutte

Reference

1. *Research Department Progress Report, August 1, 1974, p. 3*

6.4 Determination of Elemental Sulfur in Tar Sand and Bitumen

The occurrence of elemental sulfur in a tar sand might be linked to geological and chemical changes incurred during its formation. These changes may affect the processibility of a tar sand and the quality of bitumen obtained. The presence of a quantity of elemental sulfur in the synthetic crude (which has been subjected to a thermal treatment) has a bearing on its further refining.

A method has been developed (1,2) for the determination of elemental sulfur in bitumen and in tar sand which has a limit of detectability of 0.2% (based on total bitumen content). The procedure is based on the following concepts:

1. Extraction of sulfur in a suitable small fraction of the total bitumen, by the use of petroleum ether (3) over charcoal.
2. The use of ultraviolet spectroscopy where it was found that the absorption of sulfur at 262 nm is stronger than that of the selectively isolated bitumen fraction containing it. Exclusion of possible interferences was discussed (2).
3. The extraction of sulfur from the bitumen fraction with aqueous sodium sulfide (Na_2S) solution.

To summarize the analytical procedure the UV absorbances at 262 nm of selectively isolated bitumen fraction (from tar sand or whole bitumen) are measured before and after refluxing with aqueous Na_2S solution. The difference in the absorbances is correlated with sulfur concentration by referring to a pre-established calibration curve.

Future Activities

It is believed that the method can be refined to yield lower limits for sulfur detectability. The determination of elemental sulfur in bitumen and residues derived from subsequent thermal treatments, may provide a relationship between sulfur content and processibility of tar sand.

L.H. Ali

References

1. *Research Department Progress Report, October 1, 1974, p. 3.*
2. *Research Department Progress Report, November 1, 1974, p. 13.*
3. *Ali, L.H. and Al-Shahwani, K.I., "A Method for the Purification of Raw Sulfur from Bitumen and Ash Impurities". Chem Ind. (London) (in press)*

7. ANALYTICAL AND TECHNICAL SERVICES

7. TECHNICAL SERVICES

7.1 Analytical Services

7.1.1 Summary of Special Requests, 1974

The Analytical Services section provides the analytical back up for special projects and pilot scale operations. The section also handles many requests from outside the department. Table 15 summarizes work performed by the analytical group in support of these company wide activities.

The procedure for requesting special work to be carried out by the Analytical and Technical Services group has been prepared (1). This procedure has been developed for the benefit of all Syncrude personnel particularly those outside the Research Department. The procedure has been established to facilitate laboratory work requested in the future of the Environmental Field Laboratory, Reserves Evaluation Laboratory, Main Laboratory Analytical Services Group, and for Data Processing.

R.C. Shaw
C.G. Paton
R.E. Hoyle

Reference

1. *"Procedure for Handling Request(s) for Analytical Services". Syncrude Interoffice Correspondence, Ref. No. 893, R.R. Goforth to E.W. Paradowski, July 30, 1974.*

7.1.2 Syncrude Analytical Methods Manual

The revised edition of the Syncrude Analytical Methods (S.A.M) Manual was completed in the latter part of this year. As a successor to the August 1970 manual considerable augmentation was necessary to comply with the expanded scope of research activities. The inclusion of approximately forty recently developed procedures, in addition to material retained from the former edition, affords a comprehensive survey of recognized analytical methodology currently used within the Research Department.

To reflect existing research programs, the contents of the manual have been recategorized according to the system outlined below.

Table 15

Request	Date work completed	Reported by
Develop a reference table indicating process sample sizes required by analytical services (to aid samplers)	January 18	R.E. Hoyle
Analyze selected test hole samples for tar content	May 8	C.G. Paton
Flash and Fire Points on Bitumen Product from Pilot Plant Naphtha Recovery	May 22 June 4	R.E. Hoyle
Qualitatively evaluate flex characteristics of Nitrile belting at sub-ambient temperatures	Sept. 16	A.M. Scott, CBL. (R.C. Shaw)
Forward precision data applicable to O/W/S analysis of tar sand	Sept. 27	R.C. Shaw (Ref. No. 11933)
Precision of Tar Sand Analysis	Sept. 27	R.C. Shaw (Ref. No. 11933)
Suggest and evaluate alternative tar sand feed belt coatings	Oct. 1	R.C. Shaw
Sieve Analysis on Water Sand	in progress	
Soluble Salts in Tar Sand	in progress	
Evaluate resistance of Bird's ceramic adhesive to diluted bitumen product @ 170^{O}F	in progress	

Revised numerical designations for Syncrude Analytical Methods -

Section 1 - Extraction - Process Evaluation
Section 2 - Froth Treatment - Process Evaluation
Section 3 - Water Treatment - Process Evaluation
Section 4 - Bitumen Characterization
Section 5 - Diversification - Mineral Studies
Section 6 - Environmental Monitoring - Air
Section 7 - Environmental Monitoring - Water
Section 8 - Fundamental Studies

Individual methods are numbered sequentially under the applicable section. Manual content will be continually reviewed and updated as material becomes available.

7.1.3 <u>Subsampling Studies</u>

In order to minimize sampling and analysis frequencies proposed for the commercial plant extraction circuit, it is envisioned that process samples will be composited over several normal sampling periods. Consequently, direct analysis of the resultant bulk materials cannot be accommodated due to size restrictions imparted by the analytical test procedure. The design and statistical evaluation of alternative subsampling schemes is currently under investigation.

The program includes an assessment of various agitators and pretreatment steps for their ability to uniformly disperse bulk froth and tailings samples. Criteria for judging the degree of homogenization, and thus the extent to which a subsample will reflect bulk composition, have previously been defined (1).

Recent tests have been limited to the homogenization of primary froth samples with high shear impellers. The observed standard deviations for the oil, water and solids assays of consecutive subsamples withdrawn from the bulk, average, less than 2% relative. The proportion of the overall observed standard deviations contributed to by sample inconsistencies, may be derived if the precision of the analytical measurement is known. Previously reported values have been found inadequate (2) and the suggestion that analytical precision be determined on a theoretical basis is being pursued.

Future Activities

It is anticipated that further investigations will afford selection of the optimum subsampling schemes applicable to bulk process samples. Information gathered during the study will serve to better define the cumulative effect of sampling errors on the precision of analytical procedures.

R.C. Shaw

References

1. *Research Department Progress Report, September 1, 1974, p. 42.*
2. *Research Department Progress Report, November 1, 1974, p. 44.*

7.1.4 Operations Laboratory Design

The design basis memorandum for the Operations Laboratory has been completed. According to the existing schedule construction of the Operations Laboratory at the Mildred Lake Plant Site should be completed by April 1976. Mechanical completion will follow approximately 8 months later. Costs and scheduling are now being done by Carswell Engineering and the sub-contract is expected to be awarded in the fall of 1975. The philosophy behind the design basis memorandum was to design a laboratory, which would allow for maximum flexibility of personnel and equipment and the most efficient use of space at minimum cost. Using the concept, changes can be made in the interior to accept new equipment and techniques or to rearrange the old in minimum time and expense.

The laboratory will occupy 13,348 sq ft, of which 840 sq ft will be devoted to loading dock space and 2,776 sq ft to handling core hole samples. The building will be part of the administration complex.

The laboratory organization is divided into four groups; Core/Extraction, Inspection, Analytical, and Environmental. Each group will have a supervisor responsible for a number of chemists and/or technicians.

For more detailed information the "design basis memorandum" should be consulted (*references 1,2,3*).

Future Activities

At present a logic network diagram is being developed to outline future work. It includes such projects as equipment and personnel selection, methods manual, detailed interior design, data handling and pneumatic sampling delivery systems to name a few. The future development and successful start-up of the Operations Laboratory relies very heavily on a close co-operative interaction with the Research Department. Some of the more important programs which will carry well into the future include:

1. Developing subsampling techniques for extraction and froth treatment areas.
2. Faster, simpler oil/water/solids analysis.
3. Automated homogenizing of core samples.
4. Instrument evaluation, e.g., X-ray fluorescence
5. Training programs for laboratory start-up.
6. Methods development.
7. Establishment of an air monitoring network.

W.A. Foster

References

1. *Wood and Gardner Architects Ltd., "Preliminary Design Report: Laboratory", June 1, 1974.*
2. *"Operations Laboratory". Syncrude Interoffice Correspondence, Ref. No. 11274, W.A. Foster to T. Harkness, August 20, 1974.*
3. *Wood and Gardner Architects Ltd., "Operations Laboratory, Mildred Lake, Alberta; Preliminary Plan". August 26, 1974.*

7.2 Reserves Evaluation Laboratory

1973-74 Drilling Program

A new laboratory facility was put into operation in January to support an expanded drilling program. This included 95 winter core holes, 60 summer core holes, and 61 fall core holes. The winter holes have been analyzed for oil/water/solids and fines (12 holes) and the data reported to the mining group. Laboratory work is still underway on the summer and fall programs.

The 1973-74 drilling was considerably different from the past two years. Firstly, a new drilling technique was adopted using plastic pipe to encase the core. Secondly, a major summer drilling program was undertaken for the first time. In previous programs the sampling of core was carried out in an on-site laboratory using a ribbon sampling method (1) with only minimal geological descriptions. The change in drilling technique gave the geologist an opportunity to do a more extensive study of the core under good laboratory conditions. The plastic sleeve technique is being used by all operators in tar sand development.

The 1973-74 programs' laboratory support was initially on a contract basis (approximately 17 holes were completed) until the Research Department could provide facilities for the support. Because of lack of space in the main research laboratory, the reserves evaluation laboratory was set up in leased space in southeast Edmonton.

Core Handling and Laboratory Procedures

The Reserves Evaluation Laboratory procedures as presently carried out, are as follows:

1. The core is transported to the Edmonton laboratory. Each core is encased in a 2 1/2 inch O.D. PVC. pipe, which has been cut in the field into 5-6 foot intervals and capped at each end. (The summer program included refrigerated storage (maximum 40°C) in the field and delivery by refrigerated truck to minimize water losses in the tar sand.)
2. The core is frozen with dry ice in the laboratory prior to sawing.
3. The frozen 5-6 foot intervals are wet cut into 1 foot lengths, then into halves with 20", diamond blade, concrete saw.
4. The surface of one half is cleaned and passed to the resident geologist for detailed study. It is photographed and finally stored for future use.

5. The second half is sampled by dry cutting a V-notch with a carborundum blade.
6. The V-notched material is hand homogenized, sub-sampled and submitted for analysis. Eight ounce samples are retained in sealed bottles for additional analyses as required.

The above steps are carried out on a hole by hole basis. The details are documented elsewhere (2).

The laboratory has a staff of 6 laboratory assistants for the front end core handling operation, 7 laboratory technicians for analyses and one supervisor. A mining department geologist is also resident in the laboratory.

The 1974-75 Drilling Program

In addition to the 1974 fall drilling program (61 holes) it is expected that 183 core holes will be drilled in the first quarter of 1975.

Core Storage

At present all retained cores are stored in the laboratory. This function will be taken over by the Mining Department as the laboratory does not have the storage capacity beyond approximately 100 core holes or approximately 6 months of drilling. This is not adequate to store all the mining lease holes for 5 years and the exploratory holes for up to 15 years, as planned by the Mining Department.

E.W. Paradowski
R.E. Hoyle

References

1. Research Department, Annual Documentation, 1970, S-2M-70, December 11, 1970, p. 2.
2. Shaw, R.C. and Paradowski, E.W. "Reserves Evaluation Laboratory Procedures Manual" (in preparation)

7.3 Data Processing

Over the past several years, the use of computer time-sharing within the Research Department has risen dramatically and associated costs which were at one time a few hundred dollars per month had risen to several thousand dollars as of mid-1973. With this volume of computer work, the outside time-sharing was inconvenient, and often unreliable, in terms of input/output and extremely expensive. Therefore, in January, 1974, Mr. G.G. Dunbar of Imperial Oil's Systems and Computer Services Department reviewed our present and projected computing needs and provided an economic comparison, over three and five year periods, of various alternatives (1). These included time-sharing on various machines, the outright purchase of a small in-house computer and a lease-to-purchase arrangement on such a machine.

In terms of economics, the least attractive alternative was shown to be the continued use of the General Electric time-sharing service. The acquisition of a small in-house machine was clearly justified on the basis of this study (2) and arrangements were made to obtain a Digital Equipment PDP-11/40 computer on a three year lease-to-purchase plan. As acquired, this machine consists of:

32K word core memory
1.25 MM word disc
2-tape drives
high speed paper tape reader
card reader
165 character/second line printer

This particular configuration can satisfy 90 to 95% of the Department's requirements and the machine itself can be expanded to permit real time applications. The installation was completed in mid-September and presently work is in progress to convert existing programs to the new system.

K.C. Porteous

References

1. Dunbar, G.G. "Syncrude Computer Study", Imperial Oil Ltd., Systems and Computer Services Department, February 13, 1974.

2. "Research Department Computer Facilities", Syncrude Interoffice Correspondence, K.C. Porteous to R.R. Goforth, February 15, 1974.

7.4 Information Services

The section continued to seek out and supply information related to the Department's various projects by means of books, journal articles, reports, documents and patents and to arrange for their storage and retrieval.

As the Environmental Affairs Group became more functionally integrated with the Research Department, so the Research Department Information Services section became responsible for serving that group, both at its Petroleum Plaza offices and at the Mildred Lake Environmental Field Laboratory. A small collection of books and documents was initiated for that laboratory (1).

Computer produced indexes of the holdings of the Research Department Library were frequently generated. Copies were placed in the Library, Central Files, Research Office and the Environmental Field Laboratory at Mildred Lake. Furthermore, a separate print out of the index of material held at Mildred Lake was generated.

In addition, as an off-shoot of computer indexing, a bi-monthly current awareness bulletin was generated listing by most descriptive single keywords, books, documents, patents and articles indexed that period. This was distributed within the Department (2).

Commercial current awareness services particularly from the Canadian Institute for Scientific and Technical Information (CISTI) formerly the National Science Library, in Ottawa, were again actively utilized (3). In addition to Chemical Abstracts Condensates, a profile was written to be searched against the Biological Abstracts data base.

On-line, retrospective, search services against very many data bases became available this year through A.I.R.A. (Alberta Information Retrieval Association) (4). These facilities were extensively used. Later in the year it became possible to access four data bases, Chemical Abstracts, Engineering Index, Biological Abstracts, and INSPEC directly with the Department's Data comp 300 consol using the CAN/OLE system operated by C.I.S.T.I. in Ottawa.

A Carl Zeiss Dokumator microfilm reader was acquired to view core log film now deposited in the Library. A means of indexing these films is being developed. As has been customary, a copy of the core analysis is filed in the library, but in addition this year the geologic descriptions are now also retained. Copies of the electrologs are being kept for the Department's geologist.

A study was carried out on the administration of laboratory notebooks (5) for implementation in 1975.

Future Activities

Standard activities of an Information Service will be continue following the section's established procedures. The computer indexing feature will be kept up to date, with some older material, indexed in other systems, incorporated if felt relevant and useful. Particular attention will be paid to completing the indexing of all Library books and dispersing with the conventional card catalogue.

It is intended to take a more active interest in microfilm facilities. Particularly the feasibility of placing the company's formal reports on microfiche is to be investigated (6).

It is hoped that further progress will be made in expanding on-line literature searching facilities for use from within the Department.

P.J. Bates

References

1. *Research Department Progress Report, April 1, 1974, p. 23.*
2. *Research Department Progress Report, February 1, 1974, p. 42.*
3. *Research Department Progress Report, April 1, 1974, p. 24*
4. *Research Department Progress Report, March 1, 1974, p. 34.*
5. *"Laboratory Notebooks" Syncrude Interoffice Correspondence. P.J. Bates to R.E. Hoyle, August 13, 1974.*
6. *"Company Reports". Syncrude Interoffice Correspondence P.J. Bates to R.E. Hoyle, May 16, 1974.*

8. ENVIRONMENTAL STUDIES

8. ENVIRONMENTAL STUDIES

The initiation of construction activity on lease 17 during the winter of 1973-74 necessitated increased activity on the part of the Environmental Affairs Group whose specific 1974 objectives were:

1. To prevent or control adverse environmental impacts during construction and to plan for the operating phase.
2. To provide baseline information upon which changes in the natural environment brought about by tar sands developments can be evaluated.
3. To develop a better understanding of the ecosystem in which the tar sands are to be developed.
4. To report to and inform public interests.

8.1 <u>Environmental Field Laboratory</u>

8.1.1 <u>Air Quality Monitoring</u>

Syncrude's mobile air quality monitoring station has been in operation since May 1973. The air monitoring station incorporates equipment used to obtain baseline data for sulfur dioxide concentrations, hydrogen sulfide concentrations and windspeed and direction(1). Baseline studies refer to the acquisition of data to determine conditions prior to alteration or addition, specifically physical and chemical parameters of air. The air monitoring equipment includes:

1. Research Appliance Company hydrogen sulfide tape sampler, Model G 2-SE.
2. Philips sulfur dioxide monitor, PW9700.
3. Dominion Instruments wind instrument, Windflo, Model 540.

The air monitoring station is leased from Western Research and Development Ltd., Calgary, who are experienced in monitoring ambient air quality around Alberta gas plants. Western is also compiling, correlating and reporting the results in a format that is acceptable to the Alberta Department of Environment. Western calibrates the Philips sulfur dioxide monitor monthly and daily maintenance of the equipment is carried out by Syncrude Environmental Affairs personnel.

The air quality monitoring station was relocated periodically from May 1973 to October 1974 on or near the vicinity of Lease 17.

Syncrude is presently evaluating various types of ambient air monitoring equipment to assess the instruments' operability and maintenance. This will help in the selection of instrumenation for the five air monitoring stations required under the license to operate. As well as monitoring equipment presently in operation, additional instruments being evaluated are as follows:

1. Philips PW9700 Hydrogen Sulfide Analyzer
2. Meloy SA-185-2 Total Sulfur, Hydrogen Sulfide and Sulfur Dioxide Analyzer

Evaluation of instruments will continue into 1975.

Reference

1. Western Research & Development Ltd. Monthly Reports. Fort McMurray Environmental Laboratory, 1973.

8.1.2 Ground Level Meteorology

The Syncrude meteorological station is operated in conjunction with the Canadian Atmospheric Environment Service. The data obtained is forwarded to Atmospheric Canada for public and private use. Syncrude utilizes the data to determine various construction parameters which are dependent on either wind speed and direction, precipitation or temperature. The meteorological station is maintained by Syncrude environmental affairs personnel.

The meteorological station was erected in June 1973 at the Syncrude mining site and was relocated to a new permanent location on May 22, 1974. The ten meter anemometer tower has been extended to 13 meters to:

a) Eliminate interferences caused by nearby buildings, trees and overburden piles.

b) To aid in the collection of ground weather data for a cooperative Climatology Study (7.5.1).

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a) Eliminate interferences caused by nearby buildings, trees and overburden piles.

b) To aid in the collection of ground weather data for a cooperative Climatology Study (7.5.1).

Equipment operating at the meteorological station includes:

1. Recording rain gauge tripping bucket -- with associated chart.
2. Totalizing rain gauge -- records up to 10" of rain using a graduated cylinder.
3. Sun gauge -- records hours of sunlight.
4. Evaporation pan (12.3 sq. ft.) -- determines evaporation of water from a specified area.
5. Totalizing anemometer -- used with evaporation pan for ground wind speed measurements.
6. Maximum and minimum thermometers.
7. Hygrothermograph -- determines relative humidity and temperature -- 7 day chart.
8. Recording anemograph -- records wind speed and direction.

Data from the regular weather observations made daily from June, 1973 are available from the Environmental Field Laboratory.

8.1.3 Water Quality Monitoring

The surface water quality monitoring program may be structured into three areas:

1. Permit Analysis - weekly monitoring of Beaver Creek to meet Government requirements (Permit 73-WP-038).

The Permit Analysis monitoring program has been changed somewhat from the initial system in that: (a) drainage ditches as of August, 1974 are no longer being sampled. Instead, weekly samples are taken at a point upstream -- south edge of Lease #17 -- from the drainage outflows, and at a point downstream -- Beaver Creek Recreational Area -- from drainage outflows; and (b) as of October 12, 1974, the saline water discharge line has been removed from Test Pit A.

2. Baseline Analysis - systematic monitoring every six weeks at the following sites.
   - i) Upper Beaver Creek
   - ii) Lower Beaver Creek
   - iii) Poplar Creek
   - iv) Mildred Lake

v) Ruth Lake
vi) Athabasca River Dock
vii) Athabasca River Bridge

All permit and baseline analyses are available from Environmental Affairs.

3. Analysis for Special Projects - these projects have involved active participation with
   i) the Environmental Affairs Group
   ii) Renewable Resources Consulting Services Ltd. in support of a study of Ruth Lake and study of the saline effluent effects on the biota of Beaver Creek (1).
   iii) Aquatic Environment Ltd. in support of a study of the Athabasca River (2,3,4).

The Syncrude Environmental Laboratory is now capable of the following water quality analyses:

Ammonia	Hardness
Chloride	Oil and Grease
Nitrate	Chemical Oxygen Demand
pH	Conductivity
Temperature	Resistivity
Dissolved Oxygen	Threshold Odor Number
Total Phosphates	Total Solids -- 105°C
Ortho Phosphates	Total Solids -- Ignited
Carbonate	Dissolved Solids -- 105°C
Bicarbonate	Dissolved Solids -- Ignited
Phenols	Suspended Solids -- 105°C
Color	Suspended Solids -- Ignited
Turbidity	Total Organic Carbon
Sulfates	Iron
Silicates	

Syncrude Environmental Lab has followed the sampling techniques and the preservation of water samples outlined in reference (5).

J.R. Alessio
S.K. Schwarzenberger
J.W. Marchak

References

1. "An Investigation of Saline Effluent Effects on the Biota of Beaver Creek". Report by Renewable Resources Consulting Ltd. to Syncrude Canada Ltd., Fall, 1974.
2. "Proposal: Athabasca River Study - Syncrude Canada Ltd.". Aquatic Environment Ltd.
3. Letter of acceptance "Athabasca River Study". P.D. Lulman to P. McCart.
4. Letter of contract commitments. Ref. No. 6317. P.D. Lulman to P. McCart, August 8, 1974.
5. Coyne, V., "Water Sampling Guidelines and Interpretation of Data". U.S. Environmental Health Laboratory, McClellan Air Force Base, California, 1972. 50 pp. (AD-752537).

8.2 Ecological Research

8.2.1 Freshwater Biology

Aquatic habitats are visibly and rapidly affected by human activity. A study to evaluate the biological effects of diverting Beaver Creek through Ruth Lake has been in progress since March 1974.

In anticipation of the need of permits from the Alberta Department of the Environment, the discharge of saline water into Beaver Creek was evaluated for one year, beginning in September 1973.

Environmental monitoring programs and impact assessment related to the long-term disposal of saline water were initiated July 23, 1974. Subsequently, a research contract to assess the potential effects of saline effluent on the ecology of the Athabasca River, was awarded to Aquatic Environments Ltd. The consultant's program was designed with an expectation of fulfilling government requirements regarding environmental impact assessments of major developments, to provide proper engineering design data, and to ensure that deficiency statements from interested government departments are minimal. This study expected to be complete by October, 1975, is directed to five areas:

1. Assessment of the baseline conditions of the aquatic ecosystem in its present, relatively undisturbed state.

2. Selection of environmental parameters to measure change and detailed sampling of these parameters to provide a monitoring data base.
3. Assessment of the potential impact of the proposed development and recommendations concerning the course of development to ensure minimal environmental impact.
4. Survey of scientific literature relevant to various aspects of the study.
5. Studies to determine the toxicity to fish of the saline effluents particularly sensitive species such as grayling.

Preliminary aspects of the study involve, general biological inventories, test netting to determine fish collection sites, water chemistry and zoobenthos sampling. Fish toxicity studies have commenced.

In 1975, efforts will primarily involve detailed sampling of selected environmental parameters to provide a baseline for future monitoring studies. Effort will also be directed to the survey of relevant scientific literature and an assessment of the impact of the proposed development.

References

1. "A Proposal for Baseline Environmental Studies of Ruth Lake and Poplar Creek" March 1, 1974, Renewable Resources Consulting Services Ltd.
2. "An Investigation of Saline Effluent Effects on the Biota of Beaver Creek", draft report, October 1974, Renewable Resources Consulting Services Ltd.
3. "Discharge of Saline Water to the Athabasca River -- Approach for Permit Acquisition". Syncrude Inter-Office Correspondence, Ref. No. 3904, R.R. Goforth to H.B. Scott, April 11, 1974.
4. "A.F.E. No. 6431". Ref. No. 9878, R.C. Condon to Executive Committee, July 24, 1974.
5. "Proposal: Athabasca River Study -- Syncrude Canada Ltd." Aquatic Environments Ltd. to P.D. Lulman, August 1/74.
6. "Letter of Acceptance -- Athabasca River Study". Ref. No. 6000/6317. P.D. Lulman to Dr. P. McCart, Aquatic Environments Ltd., August 8, 1974.
7. Research Department Progress Report, September 1, 1974, p. 35.

8. *Research Department Progress Report, October 1, 1974, pp 37-38.*
9. *"Saline Water - Status Report". Ref. No. 17952, G.H. Robbins to List, October 25, 1974.*
10. *"Athabasca River Study - Environmental Lab Data" F/C 6510, R.E. Hoyle to G.H. Robbins, Nov. 21, 1974.*
11. *"Saline Water Disposal". Ref. No. 21715, G.H. Robbins to D.G. Adam, November 27, 1974.*

8.2.2 Ornithology

There is increasing industry and public recognition of the implications of tailings ponds to migratory birds. A major study is now underway, directed by LGL consultants, to evaluate use of water bodies by migratory waterfowl and shorebirds in the McMurray area. The data collected from four migratory periods will be utilized to develop deterrent techniques in and around tailings ponds.

References

1. *"A Revised Propsal to study the Ornithological Aspects of the Proposed Tailings Pond on Crown Lease #17 June 17, 1974, LGL Environmental Consultants Ltd.*
2. *"Letter of Acceptance - To Study Ornithological Aspects of Tailings Ponds", Ref. No. 8216, F/C No. 6940, P.D. Lulman to Dr. Schweinsburg (LGL) June 27/74.*
3. *"Workplan 1974 Studies of Syncrude Mildred Lake Tailings Pond", a proposal, LGL Environmental Consultants Ltd.*
4. *"Letter of Acceptance - To Conduct Deterrent Studies on Lower Tailings Pond", F/C No. 6315/6324, Ref. No. 10539, P.D. Lulman to Dr. Schweinsburg, August 20/74.*

7.2.3 Bio-Monitoring

Biological systems are known to vary from year to year in population (density) and species diversity. Long term investigations are essential to determine the nature of natural change as it coincides with man induced change. An entomology study was initiated this year in part, as a means of bio-monitoring long term environmental alterations. This study is to be conducted in conjunction with lichenology and soil acidification studies which are currently being funded by government agencies.

References

1. "Environmental Study - Entomology", revised proposal, May 1974, Lousier, Porter & Weseloh.
2. "Letter of Acceptance - Entomology Study", F/C No. 6304, Ref. No. 5981, P.D. Lulman to B. Porter, May 22/74.
3. "Syncrude Canada Ltd. - Entomology Study - 1974 Reconnaissance, 22 July/74-20 Aug/74" - Draft Report by Lousier, Porter & Weseloh.
4. "Atomospheric Sulphur Dioxide: effect on the pH and Sulphur content of rain & snow; and addition of sulphur to surface waters, soils & crops". Nyborg, M. and McKinnon, Allen & Associates Ltd.
5. "Effects of Atmospheric Sulpher dioxide on rain intercepted by forest vegetation", Baker, J., Hodsing D., and Nyborg M.
6. "Lichens as bioindicators of SO_2" Case, J.W.

8.2.4 Archaeology

An initial archaeological survey of lease 17 in 1973 revealed a rich resource of artifacts and indications of native Indian occupation in the McMurray area for the past 10,000 years. A second detailed "dig" was made on lease 22 in 1974 to investigate a quarry site on the banks of Beaver Creek. The report of this research will be forthcoming in December 1974 (2). During the winter of 1974, the archaeologists also made an onsite observation of the construction of 24,000 feet of a camp water supply line (3).

References

1. "A Proposal for Archaeological Investigation of the Beaver Creek Quarry Site", January 1974, submitted by Timothy Losey.
2. "The Beaver Creek Site: a Prehistoric Stone Quarry on Syncrude Lease 22". Final Report submitted by T. Losey and C. Sims.
3. "Archaeological Observation of Syncrude Lease #17 Water Pipeline Construction; A Summary Report", March 1974, submitted by Timothy Losey.

8.2.5 Revegetation

The cooperation of industry in 1973 enabled Dr. Hermann Vaartnou, Alberta Department of Agriculture, to pursue seed collection and plant species selection for revegetation purposes. Dr. Vaartnou was funded in 1974 by Syncrude to undertake detailed studies in greenhouse and field. Although continued industry cooperation through the Oil Sands Environmental Study Group, was forthcoming, revegetation research required maximum input from Syncrude in order to resolve many of the anticipated revegetation problems on Lease 17.

Reference

1. *"Establishment and Survival of Ground Cover Plantings on Disturbed Areas in Alberta", Reports 1-6, H. Vaartnou and G.W. Wheeler.*

8.2.6 Soils

Soil specialists from the University of Alberta are now working in conjunction with Dr. Vaartnou to provide soil-plant combinations most suitable in tar sand mined land reclamation. A team of five soil scientists have been commissioned since October. Preliminary soil samples have been collected from the McMurray area and are presently being analyzed and experimented with in Edmonton.

Reference

1. *"Reclamation and Revegetation of Surface Mined Areas in the Oil Sands", a soils proposal, October 1974, Rowell, McGill and Nyborg.*

8.2.7 Remote Sensing

Aerial reconnaissance through black and white colour and infrared photography was carried on again this year to produce an enviornmental record of the original condition and a record of the impact of each stage of development *(1)*.

P.G. Lee
G.H. Robbins
P.D. Lulman

Reference

1. *"Remote Sensing and the Athabasca Tar Sands: an Overview". Edmonton, 1974. 50 pp. Environmental Research Monograph, 1974-1.*

8.3 Publications

In a continuing effort to maintain public and employee awareness of Environmental activities, several issues of the "Environmental Bulletin" have been published. A poster series is being distributed periodically throughout Syncrude and Bechtel work areas to inform employees of ongoing environmental programs. The latest monograph "Remote Sensing and the Athabasca Tar Sands" *(1)* is now being distributed to a wide public and scientific audience. Two further monographs, concerned with revegetation research and archaeology respectively are in the final stages of preparation for publication.

J.A. Wright

Reference

1. *"Remote Sensing and the Athabasca Tar Sands: An Overview" Environmental Research Monograph 1974-1, Syncrude Canada Ltd.*

8.4 Environmental Engineering

8.4.1 Upper Atmosphere Meteorology

A cooperative regional meteorological study has been established in the Athabasca tar sands region. The participating operators are Shell, GCOS, Fina, Home-Alimex, and Syncrude. The MEP Company has been retained as a consultant in the development of a regional meteorological model.

The objectives of the study are to provide and verify data for stack design through determination of adiabatic lapse rates and dispersion climatology. The results should also act as a guide to the selection of sites for ambient air monitoring stations and static exposure cylinders required under the air permit to construct.

Twice daily mini-sonde releases will provide information regarding atmospheric wind speeds and temperature profiles. Mini-sonde releases from Shell's lease commenced at the end of September. A parallel program will begin on Lease 17 as soon as all required equipment has been delivered. For adverse weather conditions, plans are in progress to release mini-sondes from Fina, Home and other Syncrude leases. The present program is for the duration of one year and should terminate in October, 1975.

References

1. *"Meteorology Data Analysis Program". Letter to R.B. Gorby, Shell Canada Ltd. from T. Voksepp, Ref. No. 16693, October 23, 1974.*
2. *"Expanded Tar Sands Meteorological Analysis Program". Copy of letter from P.J. Benn, Shell Canada Ltd. to M.S. Hirt, MEP Company, December 5, 1974.*
3. *"Addition to proposal re: Lease 13 (June, 1974) - An expansion program to include, Home, Petrofina, and Syncrude". Letter to R.B. Gorby, Shell Canada Ltd. from M.S. Hirt, MEP Company, Syncrude Ref. No. 15560, September 27, 1974.*
4. *Proposal to Shell Oil Ltd Tar Sands Project. Dispersion of proposed Effluent Emissions, prepared by the MEP Company, June, 1974.*

8.4.2 Tar Sands Air Monitoring Network

A preliminary study has been completed in an attempt to establish a combined ambient air monitoring network for operators of tar sands plants. Participants in such a network could consist of Shell, Fina, Home-Alminex, GCOS and Syncrude. The concept of a combined network has been accepted in principle by all potential operators and representatives of the Alberta Department of the Environment (DOE) have encouraged the approach. Some of the major advantages of such a network would be:

1. standardization of equipment
2. simplified maintenance and reduced operating costs
3. development of a simplified, standardized and automated reporting procedure to the DOE
4. possible reduction in the number of stations required and/or protection against future increased requirements

Future work will be required in order to establish a detailed work plan and to update the preliminary study. Additional contacts with the DOE will also be required to investigate the possibility of reducing the number of monitoring stations as originally outlined in the "Oil Sands Criteria". The implementation of such a network should yield valuable information on dispersion of air emissions and should also meet provincial government permit requirements.

References

1. "Tar Sands Air Monitoring Network" J.W. Marchak, W.A. Foster, T. Voksepp, September, 1974.
2. "Minutes of Meeting, Tar Sands Air Monitoring Network". Syncrude Interoffice Correspondence, Ref. No. 17958, T. Voksepp to R.R. Goforth, November 7, 1974.
3. "Tar Sands Air Monitoring Network". Letter to J. Lack, Pollution Control Division, Alberta Dept. Environment from T. Voksepp, Ref. No. 17953, October 29, 1974.

8.4.3 Water Balance

The all-embracing term "water balance" was during the early part of 1973 identified by the now disbanded Environmental Task Force as the most critical environmental problem. The status of the questions raised by the Task Force were summarized

during May, 1974 (1). During November, 1973, Bechtel issued a report dealing with the build up of impurities in the water system (2). Bechtel has also prepared a report (3) presenting models showing the theoretical behaviour of the tailings pond and the quantities of reclaim and other waters available from startup to the end of year 2002.

Presently work is in progress and will continue in an attempt to establish a computerized modular material balance for the predictive behaviour of the water system. In the future, as additional data with regards to the inorganic mineral content of the tar sands and mine formation waters becomes available, the water balance will be recalculated in order to establish as closely as possible the expected impurities levels in the various water containing streams.

T. Voksepp

References

1. "Status of Water Balance" Syncrude Inter-office Correspondence, Ref. No. 5822, May 16, 1974, T. Voksepp to R.R. Goforth.
2. "Estimate of Accumulation of Dissolved Impurities and Water Balance - Plant 39", Canadian Bechtel Limited, November 1973.
3. "Water Sources, Consumption and Distributions" Canadian Bechtel Limited, February, 1974.
4. "Regional Hydrogeological Study, McMurray Oil Sands Area, Alberta: phase 1". J.C. Sproule and Assoc. for the Oil Sands Environmental Study Group, October, 1974.

8.5 OSESG

The OSESG (Oil Sands Environmental Study Group) was formed in 1973 and has grown to current membership of 23 oil companies. The objective of the group is to develop, by coordinating, engaging in, promoting and/or sponsoring environmental research programs, the information necessary to evaluate the environmental impact of development arising from and related to the development of the oil sands.

To date, two projects have been commissioned and completed by the OSESG: a revegetation study program conducted by Dr. Hermann Vaartnou of the Alberta Department of Agriculture; and a hydrogeological study program coordinated by J.C. Sproule & Associates.

There is another research program about to be commissioned which will study the effects of SO_2 (sulphur dioxide) on vegetation to aid in defining acceptable limits for SO_2 emissions.

In the spring of 1974, it became apparent that industry would be expected by the federal and provincial governments to make a major contribution toward environmental data collection, studies, research and control. In the fall of 1974 a relatively close working relationship was established by the Federal and Provincial governments for research in the environmental phases of the tar sands area. It was clear that both levels of government were looking to the OSESG to represent industry.

Environmental research by government agencies in the tar sands area is anticipated but remains unspecified. Industry participation in the environmental research planning has been minimal. Management of the research is carried out by OSERP (Oil Sands Environmental Research Program) within the Alberta Department of the Environment. Direct representation of industry on OSERP subcommittees is made by appointed members of OSESG. Syncrude Environmental Affairs represents OSESG on the Vegetation, Terrestrial Fauna and Land Use Subcommittees. Clarification of government expectations for industry financial support to OSERP is expected in the near future.

P.G. Lee

References

1. "Oil Sands Environmental Study Group: Status Report" July 1974, Syncrude internal report prepared by Peter Lee.
2. "Regional Hydrogeological Study: McMurray Oil Sands Area, Alberta" prepared for the OSESG October 1974, J.C. Sproule & Associates Ltd.
3. "Proposal - Field Experimentation Study on Effects of SO_2 on Vegetation". Letter from H. Becker to Dr. Legge, Aug. 22/74.
4. "Letter of Acceptance - SO_2 Stress on Vegetation" - Letter, Ref. No. 14844, P. Lee to H. Becker, Oct. 8/74.

9. D I V E R S I F I C A T I O N

9. DIVERSIFICATION

9.1 Heavy Mineral By-Products

1. Mineral Beneficiation

A process for upgrading heavy minerals from the tar sands, more particularly from the Bird scroll centrifuge tailings stream, will include the following sequence of stages: (a) removal of hydrocarbon from the tailings; (b) gravity beneficiation of the tailings by means of hydroclone, Humphreys spiral, Reichert cone, etc., and (c) separation of zircon from titanium minerals using high tension and magnetic separation techniques.

a) Hydrocarbon Removal

Fabrication of a 6 inch diameter fluidized bed reactor for burn off of scroll tailings (in 25 lbs. batch size) was completed early in 1974. Removal of the bitumen from the tailings in the fluidized bed (2) is accomplished in two stages, (i) by thermal cracking in an inert atmosphere while the bed temperature is being raised to 1025°F and (ii) by the exothermic burn off of the residue of coke (char) with air or an air-nitrogen mixture at bed temperatures ranging from 1000° to 1400°F.

b) Gravity Beneficiation

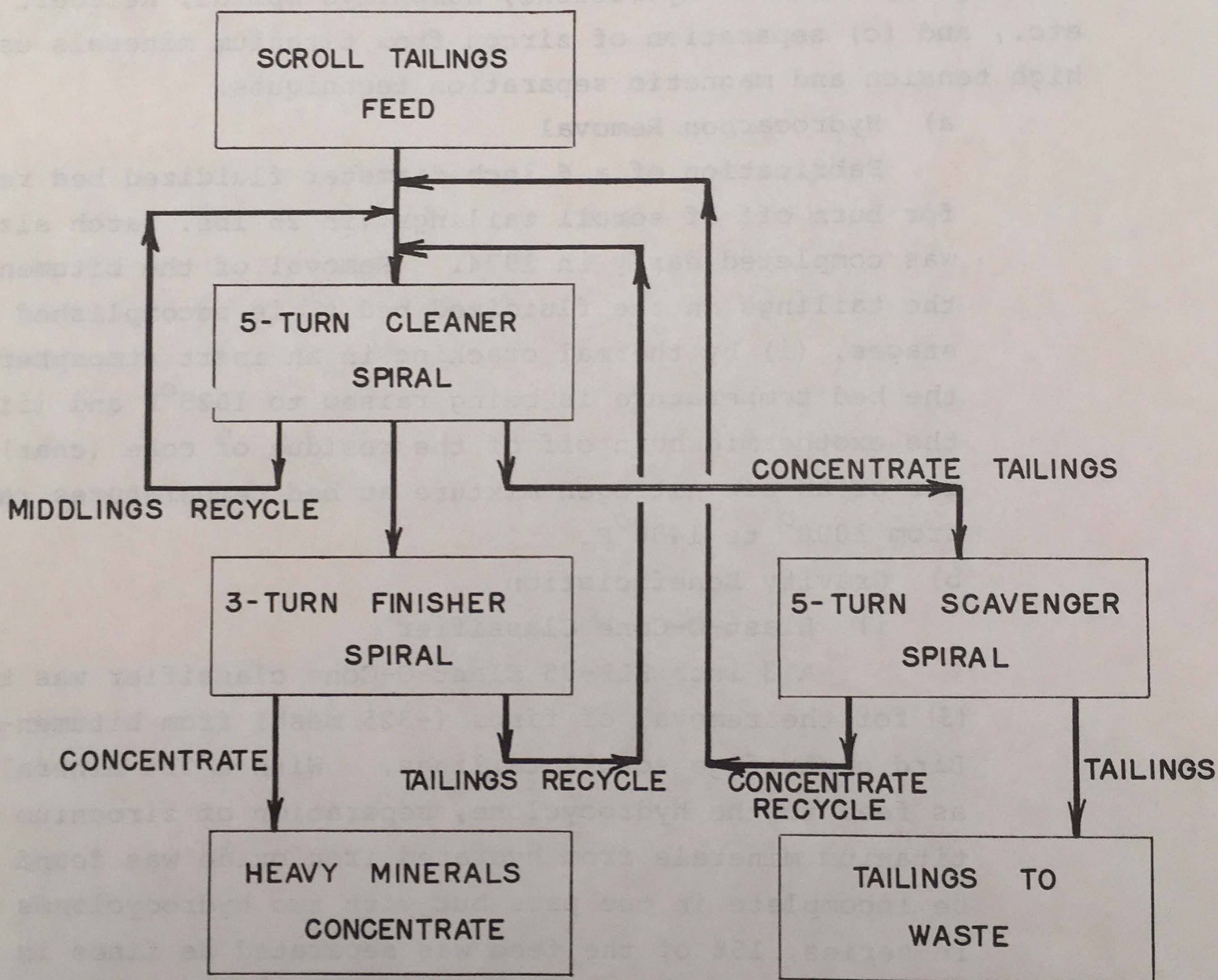
i) Elast-O-Cone Classifier

A 3 inch ELP-95 Elast-O-Cone classifier was tested (3) for the removal of fines (-325 mesh) from bitumen-free Bird centrifuge scroll tailings. With a 25% mineral slurry as feed to the hydrocyclone, separation of zirconium and titanium minerals from hydrated iron oxide was found to be incomplete in one pass but with two hydrocyclones operated in series, 15% of the feed was separated as fines in the overhead stream and 95% of the titanium and essentially all of the zirconium was recovered in the underflow from the two hydrocyclones.

ii) Humphreys Spiral

The Humphreys spiral has been thoroughly tested for use in gravity beneficiation of titanium and zirconium heavy minerals. Initial tests (4) were conducted using

FIGURE 34


SCROLL TAILINGS FEED
5-TURN CLEANER SPIRAL
MIDDLINGS RECYCLE
CONCENTRATE
TAILINGS
3-TURN FINISHER SPIRAL
5-TURN SCAVENGER SPIRAL
CONCENTRATE
TAILINGS RECYCLE
CONCENTRATE RECYCLE
TAILINGS
HEAVY MINERALS CONCENTRATE
TAILINGS TO WASTE

the spiral in a closed circuit mode. Following the construction of a 156 gallon vessel which fed a mineral slurry (25% pulp density) continuously to the gravity separator it was possible to investigate a 3 spiral circuit (cleaner, finisher, and scavenger spirals - see Figure 34) for upgrading the heavy minerals in scroll tailings (5). Recovery efficiency in the 3 spiral circuit was 70 to 80% for titanium and 90% for zirconium. (6)

c) Electrostatic Separation

The concentrate obtained from gravity beneficiation will be suitable for further upgrading by high tension and magnetic separation techniques. As a first step in this direction preliminary trials were conducted using a Carpco "Research Model" high tension separator.

2. Distribution of Heavy Minerals in Tar Sand

A study has recently been initiated to determine the distribution of heavy minerals in tar sand samples of varying grade and composition. A suitable drilling core was selected and individual samples were processed using the stainless steel pot in order to concentrate heavy minerals in the froth which was then analyzed for titanium, zirconium and iron. The determination of titanium levels was sufficiently accurate to provide a heavy minerals profile as a function of depth in the oil sand deposit.

3. Market Potential

Several companies engaged in the heavy minerals business have expressed an interest in the titanium and zirconium potential of the tar sands during recent months. Du Pont (8), Canadian Titanium Pigments Limited (N. L. Industries) (9) and Consolidated Gold Fields Limited have indicated their interest in Syncrude heavy minerals. The potential for zirconium metal production in Canada and the United States was discussed with representatives of Eldorado Nuclear Limited and Wah Chang Corporation (Teledyne) (10).

4. Clay Recovery

Tailings streams from the extraction and D.C. pilot plants were examined in the laboratory to determine the quality of clay which may be recovered. Analysis of a typical clay sample (11) showed 8% aluminum, 2% iron and 32% silicon. Samples of tailings sand or clay were supplied to Alberta firms conducting tests on (1) the production of foamed concrete precast building panels and (2) ceramic tiles.

Future Activities

It is planned to continue work directed towards the development of a process for recovery of titanium and zirconium values from scroll centrifuge tailings. Emphasis will be placed on the use of high tension and magnetic separation techniques for upgrading concentrates prepared in the Humphreys spiral test circuit. Further analysis of heavy minerals in drill cores will be conducted in order to better define heavy minerals potential from Lease 17. Contacts with potential users of zirconium and titanium minerals will be maintained.

The opportunities for commercial development of other by products will be further evaluated. The by product of interest will include ammonia, nickel and vanadium from coke or fly ash, petrochemicals such as ethylene, propylene, aromatics, V.C.M. etc., and sand and clay.

L.W. Trevoy

References

1. *Research Department Progress Report, May 1, 1974, p. 24.*
2. *Research Department Progress Report, August 1, 1974, p. 6.*
3. *Research Department Progress Report, June 1, 1974, p. 26.*
4. *Research Department Progress Report, July 1, 1974, p. 11.*
5. *Research Department Progress Report, October 1, 1974, p. 17.*
6. *Research Department Progress Report, December 1, 1974, p. 32.*
7. *Research Department Progress Report, April 1, 1974, p. 17.*
8. *"Visit of Du Pont Representatives - Minerals Development". Syncrude Interoffice Correspondence, Ref. No. 15004, L.W. Trevoy to R.R. Goforth, October 4, 1974.*

9. "Visit of N. L. Industries, Inc. and Canadian Titanium Pigments Limited Representatives - Minerals Development". Syncrude Interoffice Correspondence, Ref. No. 15005, L.W. Trevoy to R.R. Goforth, October 22, 1974.

10. "Zircon and Raw Material for Zirconium Metal Production", Syncrude Interoffice Correspondence, Ref. No. 11930, L.W. Trevoy to R.R. Goforth, September 6, 1974.

11. Research Department Progress Report, March 1, 1974, p. 29.

9.2 Caustic and Chlorine Production Study

A report (1) entitled "A Study of the Feasibility of Chlor-Alkali Production in Connection with Oil Sands Development at Fort McMurray" was issued. The report proposed integration of the Syncrude synthetic crude oil plant with a chlor-alkali production plant and a vinyl chloride monomer (VCM) synthesis unit. Caustic soda production would be supplied to oil sands plants in the Fort McMurray area. In order to provide a market for chlorine coproduced, ethylene extracted from the off-gas from the fluid coking of bitumen would be utilized and reacted with chlorine to produce VCM.

A preliminary economic evaluation (2) of chloralkali and VCM production indicated discounted rates of return of 26.7% for chlor-alkali and 30.1% for vinyl chloride monomer production based upon chloralkali production expanding from 76.4 M tons per year to 114.6 M tons per year and VCM staged production expanding from 114 M lbs per year to 171 MM lbs per year.

L.W. Trevoy

References

1. Martin, H.M. "A Study of the Feasibility of Chlor-Alkali Production in Connection with Oil Sands Development at Fort McMurray", January, 1974.

2. "Preliminary Economic Evaluation of Chlor-Alkali and Vinyl Chloride Monomer Production". Syncrude Interoffice Correspondence, F.J. Werth to L.W. Trevoy and R.R. Goforth, January 22, 1974.

3. Letter from R.R. Goforth to Executive Committee, January 21, 1974.

10. R E S E A R C H O P E R A T I O N S

10. RESEARCH OPERATIONS

10.1 Laboratory Experimental Extraction Circuit

With the completion of the engineering of the commercial extraction unit design, the need for the extraction piloting will diminish, but a significant portion of continuing and future research and development activities will be related to the hot water extraction process itself or some stream from this process such as froth, middlings or tailings. Irreversible changes are caused by storage of these materials over relatively short periods of time, which lead to spurious test results. Therefore, an extraction unit is a necessity in terms of both extraction research and the production of feed streams for other test work.

The existing 15 TPH extraction pilot plant is expensive to operate and the quantities of sand, water and froth present some severe logistics problems. These difficulties are a direct result of the 15 TPH tar sand feed rate. With properly designed equipment it will be possible to generate meaningful extraction data with a circuit processing 2.5 tons of tar sand per hour. This particular feed rate translates to both realistic vessel, line and pump sizes.

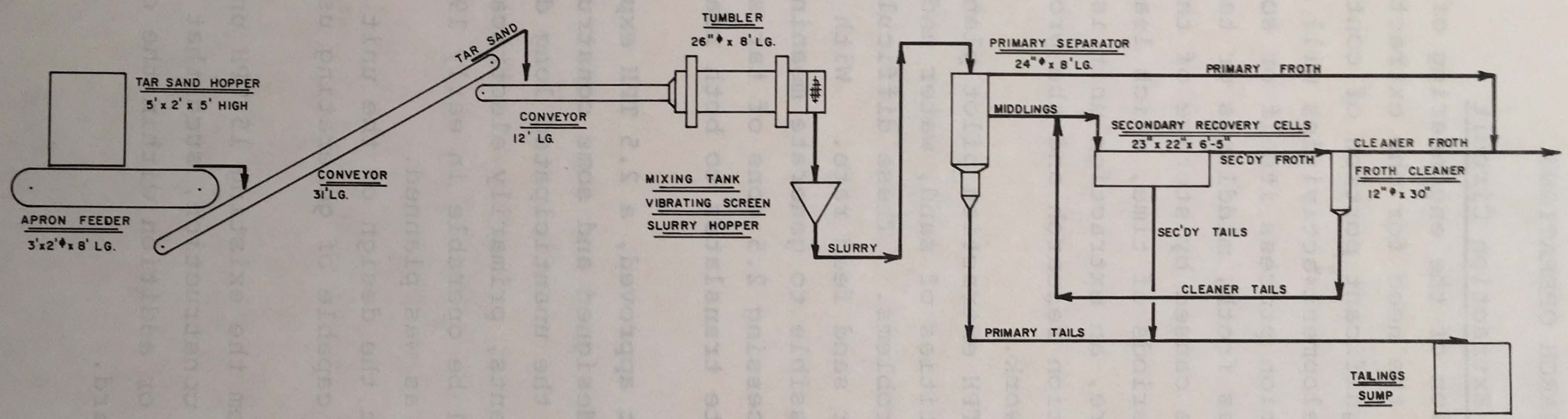
With this concept approved, a 2.5 TPH experimental extraction circuit was designed and some construction work initiated. Because of the unanticipated long delays in the delivery of some components, primarily electrical and instrumentation, the circuit will be operable in early 1975 rather than the latter part of 1974 as was planned.

The key points in the design of the unit are:

1. Minimum size capable of generating useful engineering design data.
2. Scaledown from the existing 15 TPH unit.
3. Modular type construction, such that equipment substitution or addition within the circuit is straightforward.

FIGURE 35.

LABORATORY EXPERIMENTAL EXTRACTION CIRCUIT



STREAMS: NUMBER	1	3	5	7	9	11	13	15	17a	17b	19	21	23	25	27	29	31	33	35	37	39	41	43
NAME	TAR SAND FEED	STEAM	STEAM LOSS	SLURRY WATER	CAUSTIC	SLURRY	SCREEN SPRAY	REJECT	FLOOD WATER	FLOOD WATER	GLAND WATER	PUMPED SLURRY	UNDER-WASH	PRIMARY TAILS	PRIMARY FROTH	MIDDLINGS	SEC'DY FEED	SEC'DY FROTH	CLEANER TAILS	CLEANER FROTH	COMB'. FROTH	SEC'DY TAILS	COMB'. TAILS
TEMPERATURE - °F																							
SPECIFIC GRAVITY																							
FLOWS: TOTAL LBS./HR.	5000	121	$50.83\dot{3}$	1000		6070.16	550	160	1666.6		16.6	8143.5		5112.5	$793.\dot{3}$	$2237.\dot{6}$	2268.3	99	30.66	68	861.3	2169.6	7632.16
USGPM																							
$FT.^3$/HR.																							
OIL LBS./HR.	567	0	0	0		567	0	$6.89\dot{6}$	0		0	560.103		$20.961\dot{6}$	$501.30\dot{6}$	37.835	$38.331\dot{6}$	$29.238\dot{3}$	.496	$28.408\dot{3}$	529.715	$9.42\dot{6}$	$30388\dot{3}$
WATER LBS./HR.	256	121	$50.83\dot{3}$	1000		$1326.1\dot{6}$	550	$15.42\dot{3}$	$1666.\dot{6}$		$16.\dot{6}$	3544.76		1607.37	$227.68\dot{6}$	3209.02	17131.435	$54.39\dot{6}$	22.415	$31.981\dot{6}$	$259.668\dot{3}$	$1677.038\dot{3}$	$3649241\dot{6}$
SOLIDS LBS./HR.	4177	0	0	0		4177	0	137.68	0		0	4039.32		$3484.168\dot{3}$	64.34	$490.81\dot{6}$	498.56	15.365	7.755	7.61	71.95	4832016	3967.37
ASSAYS: WT. % OIL	11.34	0	0	0		9.34	0	4.31	0		0	6.88	0	0.41	63.19	1.69	1.69		1.62	41.78	61.50	0.44	0.40
WT. % WATER	5.12	100	100	100		2185	100	9.64	100		100	43.52	0	31.44	28.70	76.38	76.33		73.09	47.03	30.15	77.29	47.62
WT. % SOLIDS	83	0	0	0		68.81	0	86.05	0		0	49.60	0	68.15	8.11	21.93	21.98		25.29	11.19	8.35	22.27	51.98
100 S/O																							
100 W/O																							

4. Totally independent, in terms of feed, control and product systems, such that the existing 15 TPH circuit remains operable.
5. Construction such that it can be easily moved to the new research facility.

The basic stages in tar sand processing (feeding, slurrying, and extraction) have been isolated, utilizing as much of the existing building walls as possible. In this manner, not only a better operating environment is achieved, by separating the cold, hot and steam areas, but the "dirty" areas do not overlap the "clean".

Initially, such items as sampling and weighing of streams will remain as manual functions. However, the design allows for installation of automatic equipment with the minimum of difficulty.

A flow sheet (diagramatic) and material balance of the circuit is shown in Figure 35. The listed vessel and equipment sizes are for the design feed rate of 2.5 TPH, but the design contains sufficient flexibility to allow for feed rates up to 4 TPH if necessary.

G.R. Lorenz

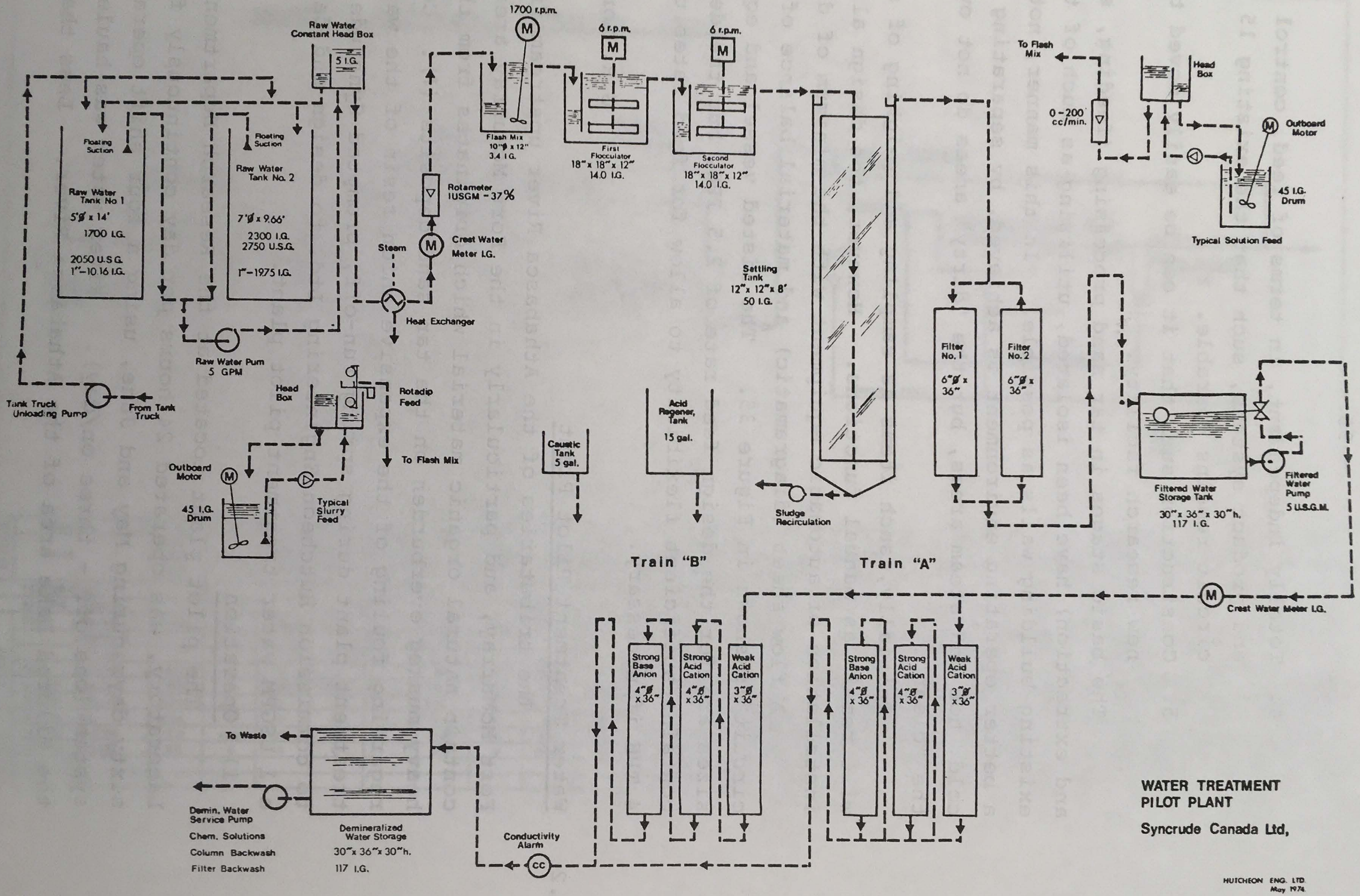
0.2 Water Treatment Pilot Plant

The tributaries of the Athabasca River upstream from Fort McMurray, and particularly in the Fort McMurray area, contain natural organic material which originates from the heavy muskeg overburden on the tar sand deposits (1). Concern regarding fouling of the expensive anion resin of the water treatment plant during spring run-off, prompted Syncrude Engineering to commission Hutcheon Engineering Ltd. to design and fabricate a 1 USGPM water treatment pilot plant.

i) Operation

The pilot plant located at the Research Department Laboratory, was operated 24 hours per day continuously for sixty days during May and June, using a four shift operating system (one off - three on/day). River water was hauled from the Mildred Lake area of the Athabasca River. Less than one

FIGURE 36



Raw Water Constant Head Box
5 I.G.
1700 r.p.m.
6 r.p.m.
6 r.p.m.
To Flash Mix
Head Box
0-200 cc/min.
Outboard Motor
45 I.G. Drum
Typical Solution Feed
Floating Suction
Floating Suction
Raw Water Tank No. 2
7'Ø x 9.66'
2300 I.G.
2750 U.S.G.
1"-19.75 I.G.
Raw Water Tank No 1
5'Ø x 14'
1700 I.G.
2050 U.S.G.
1"-10.16 I.G.
Flash Mix 10"Ø x 12" 3.4 I.G.
First Flocculator 18"x 18"x 12" 14.0 I.G.
Second Flocculator 18"x 18"x 12" 14.0 I.G.
Rotameter IUSGM -37%
Crest Water Meter I.G.
Steam
Heat Exchanger
Raw Water Pum 5 GPM
Settling Tank 12"x 12"x 8' 50 I.G.
Filter No. 1 6"Ø x 36"
Filter No. 2 6"Ø x 36"
Tank Truck Unloading Pump
From Tank Truck
Head Box
Rotadip Feed
To Flash Mix
Outboard Motor
45 I.G. Drum
Typical Slurry Feed
Caustic Tank 5 gal.
Acid Regener. Tank 15 gal.
Sludge Recirculation
Filtered Water Storage Tank 30"x 36"x 30"h. 117 I.G.
Filtered Water Pump 5 U.S.G.M.
Train "B"
Train "A"
Crest Water Meter I.G.
Strong Base Anion 4"Ø x 36"
Strong Acid Cation 4"Ø x 36"
Weak Acid Cation 3"Ø x 36"
Strong Base Anion 4"Ø x 36"
Strong Acid Cation 4"Ø x 36"
Weak Acid Cation 3"Ø x 36"
To Waste
Demin. Water Service Pump
Chem. Solutions
Column Backwash
Filter Backwash
Demineralized Water Storage 30"x 36"x 30"h. 117 I.G.
Conductivity Alarm
CC
WATER TREATMENT PILOT PLANT
Syncrude Canada Ltd,
HUTCHEON ENG. LTD. May 1974

week after start-up, the river "broke-up", which allowed for only a short base line operation before the break up water arrived. Spring break up also necessitated daily half truck loads of water because of the weight restrictions on the highways.

As well as Syncrude laboratory analysis and external laboratory analysis, the operators performed many minor quick analyses from which they determined operating parameters. The operators also kept detailed records of observations.

The pilot plant flow sheet is shown in Figure 36.

ii) Analytical Support

The analytical work was split three ways:

1. Information required for on-line adjustments was obtained by the operators.
2. High volume analyses were carried out by the analytical laboratory where they did not fall under 1.
3. Low volume analyses were contracted to Chemical and Geological Laboratories.

The analytical frequency and schedule of analyses, as scheduled at the start of the program, are shown on Table 16 .

It was found that the cycle composite analysis for silica was deceptive, since the silica content was low during the first part of the run and high during the last part (2).

The water treatment pilot plant shut down on June 14. At that point there had been 168 resin cycles sampled for analysis with each cycle ranging from one to nine sampling intervals. The average would be between two and three. Each sampling interval required from 7 to 11 individual determinations. In addition, a number of determinations were done once per cycle, or at other intervals.

iii) Conclusions

The function of Syncrude Research in this program was to operate the pilot plant and do the analytical work. For details of the design philosophy and parameters, results, observations and conclusions reference is made to the Hutcheon Engineering Ltd.

report (1). However, the following extract from that report summarizes the conclusions.

1. The pilot water treatment plant indicates that irreversible organic fouling of the anion exchange resin in the proposed demineralizer installation is a significant problem. One resin test sample failed in the equivalent of one month operating time projected to the actual plant. Significant loss of capacity was evident when 50,000 gallons of water were processed per cubic foot of resin.
2. The organic content of the water cannot be reduced to an acceptable level with the proposed pretreatment plant operated either as a clarifier or as a cold lime softener. The use of various coagulants and coagulant aids did not significantly improve the organic removal from the water supply.
3. Provision should be made at this time to incorporate addition or revised equipment to reduce the organic loading or to tolerate the organic loading which will be encountered. There is not an optimum method of ensuring complete removal of organic material at this time, and further evaluation would be required.

Syncrude Engineering has indicated that another fairly extended pilot plant run may be required following spring break up 1975 (3). The pilot plant has been crated and stored and will be reassembled if further work is necessary. It should be noted that some of the analytical equipment used in these programs has been transferred to the Environmental Field Laboratory.

G.R. Lorenz
G.R. Thompson

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2. *Research Department Progress Report, June 1, 1974, p. 21.*
3. *"Water Treatment Pilot Plant" Syncrude Interoffice Correspondence, Ref. No. 14045, G.R. Gray to N. Malychuk, Sept. 17, 1974.*

10.3 Safety Report

(Syncrude Research Department Staff, December 1, 1973 to November 30, 1974). Hours of exposure for injury experience for Research Department permanent staff only.

Hours since last lost time event (May 8, 1972 to November 30, 1974)	301,674.75
Hours worked in 1974 without lost time (December 1 to 31, 1973)	141,890.75
Hours of work since last medical aid accident	42,495.75
Number of reports of medical aid accidents (December 1, 1973 to November 30, 1974)	4

G.F. Davidson

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12. DIRECTORY OF STAFF

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In addition the Research Department retains 6 hourly/contract and 9 temporary personnel, bringing the total staff to 104 as of December 31, 1974.